THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

MARCH, 1956

Page

General

Vacuum Melting of Metals and Alloys 28
Reactor Materials and Design 28
Analysis of Thin Films 29
Determination of Metals in Oils and Petroleum Products 29

Nickel

Nickel: Supply and Demand in the U.S.A. 30
Measurement of Thermal Conductivity of Nickel 30
Sorption of Carbon Monoxide on Nickel 30, 31
Adsorption of Hydrogen and Carbon Monoxide by Nickel 31
Diffusion of Hydrogen in Nickel 31
Nickel Catalysts: Thermomagnetic Analysis 31
Gravimetric Determination of Nickel 31
Exchange Reactions of Metal Oxides 32
Extraction of Trace Amounts of Nickel from Cobalt Salts 32
Toxicity of Nickel Carbonyl 32
Growth of 'Whiskers' from Metal Salts 32

Electrodeposition and Other Coating Methods

Deposition of Metals and Related Subjects: Bibliography 32
Automatic Nickel/Chromium Plating of Automobile Components 32
Bright Nickel-Plating Process of High Levelling Capacity 33
Structure of Electrodeposited Nickel 33
Resistance of Nickel/Rhodium-Plated Parts to Moist Ozone 33
Specifications Relating to Electrodeposited Coatings 33
Production of Diffused Nickel–Cadmium Coatings 34
Electroless Nickel Plating : German Experiments 35
Electroless Nickel Plating of Aluminium 35
Metal Spraying: Recommended Procedure 35
Nickel-Plated Steel Sheet and Plate: 'Lectroclad' 35
Effects of Metal-Finishing Effluents on Sewage Purification 36

Non-Ferrous Alloys

Inter-Diffusion of Copper and Nickel 36
Copper–Nickel–Chromium Alloys: Constitution 37
Nickel–Rhenium Alloys 37
Nickel–Tellurium Alloys 37
Nickel–Zirconium Alloys 37
Anelastic Phenomena in Gold–Nickel Alloys 37
Nickel-containing Copper–Tin Bronzes 38
Properties of Age-Hardenable Aluminium–Nickel–Copper Alloys 38
Nickel Silver in Watches 38
Joining of Nickel and Nickel Alloys 39

Page

Nickel-Iron Alloys

Slip Patterns in Deformed Nickel and Nickel–Iron Alloy 39
Magnetic Domains in Nickel–Iron Films 39

Cast Iron

Specification for S.G. Iron 39

Constructional Steels

Nickel–Molybdenum Die Steel 39
Nickel–Alloy Steel Gears 40
Nickel-containing Materials in Marine Engineering 40
Specification for Steel Plate, Sheet and Strip 40
Transformations in Nickel–Alloy Steel in Relation to Weldability 41
Heat-Treatment of Welded Alloy Steels 41

Heat- and Corrosion-Resisting Materials

Precision Forging of High-Nickel Alloy Turbine Blades 42
Nickel-containing Alloys in Aircraft Gas Turbines 42
Creep-Rupture Properties of 18-8 Chromium–Nickel Steels 42
Influence of Environment on Behaviour of Metallic Materials at High Temperatures 43
Clad Molybdenum Sheet: Production and Properties 47
Sprayed Metal Coatings for Protection of Molybdenum 48
Oxidation of Stainless Steels 48
Determination of Susceptibility to Attack by Vanadium and Sodium Compounds 48
Oxidation of Nickel–Chromium Alloy Wires in Thermocouples 49
Electric-Furnace Production of Stainless Steel 49
Structure and Properties of 25–12 and 25–20 Chromium–Nickel Steels 50
Corrosion by Hydrogen Sulphide at Elevated Temperatures 51
Descaling of Steels and Alloys after Exposure to High-Temperature Water 51
Cleaning of Stainless-Steel Food-Service Equipment 52
Monel Buckets in Salt Driers 53
Influence of Oxygen on Corrosion of Stainless Steel in Sodium 53
Corrosion of Car Bodies: Resistance of Plain and Alloy Steels 53
Stainless-Steel Equipment in Production of Pharmaceuticals 53
High-Manganese Stainless Steels 53
Nomograph for Calculation of Corrosion 54
Nickel–Alloy Hard Facing on Compressor Rods 54

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Vacuum Melting of Metals and Alloys

F. T. CHESNUT: 'Vacuum-Melted Metal: An Interim Report.'
Metal Progress, 1955, vol. 68, Dec., pp. 118–23.

The major portion of the material in this article is extracted from a paper read at the National Metal Congress in 1955.

It is stated that a survey of current practice indicates that 'although much experimentation (on vacuum melting) is under way, the consensus of opinion appears to be that actual requirements can still be met by careful design and fabrication of alloys melted by more conventional methods'. It is, however, generally agreed that properties are likely to be improved in many cases by use of vacuum technique, and the potential value of the process is being tried out for materials in which, from an economic point of view, the extra expenditure involved is likely to be justifiable in large-scale production. As a support to this general conclusion, the author gives details of experience in individual works in which tests of vacuum melting are being made. The reports made relate to production of high-temperature alloys of nickel and cobalt base for jet-engine blades, magnetic alloys of the aluminium-iron type and antifriction bearings.

It appears clear that although for some materials vacuum melting offers unquestionable advantages with regard to control of composition and enhancement of properties, for other alloys the benefits are not so clearly defined, and in all cases many factors, especially economic considerations, must be weighed before the ultimate prospects of the process can be correctly assessed.

Reactor Materials and Design

U.S. ATOMIC ENERGY COMMISSION: 'Reactor Handbook.' Published, in 6 vols., by *U.S. Atomic Energy Commission; McGraw-Hill Book Co., Inc., New York, Toronto, London*, 1955.

The rapid advance of reactor technology and its increasing application to industrial power reactors have given rise to an urgent need for comprehensive review and critical correlation of nuclear-engineering data. To meet this need, the Reactor Development Section of the U.S. Atomic Energy Commission has organized compilation, by a Review Board, of a Reactor Handbook, which is issued in six volumes dealing with specific aspects of the subject.

The work of the Board responsible for the Handbook has been critically reviewed by specialists associated with the nuclear laboratories of the most important U.S. organizations operating in this field, and the material presented is thus an authoritative selection of reference data.

The titles of the Handbook sections now published are shown below:—

Research Reactors.
Reactor Handbook: Physics.
„ „ : Engineering.
„ „ : Materials.
Neutron Cross Sections.
Chemical Processing and Equipment.

The Materials and Engineering Sections contain information of major interest in relation to nickel and nickel-containing materials.

'Reactor Handbook':
'Materials: General Properties'; 610 pp. Price £3.10.0.

The main object of the group responsible for the compilation of this volume has been to summarize material useful to metallurgists, ceramists and other workers in the field of materials, and attention has also been given to making the data useful to workers in other fields of reactor technology. It is emphasized that selection of information has presented some difficulties, and that the treatment of the materials covered varies from one case to another. For example, in the case of uranium or zirconium the information is culled almost entirely from A.E.C. sources, and is relatively complete, whereas for stainless steel, for which a large volume of information exists in the open literature, highly critical selectivity has had to be exercized, with particular reference to data likely to be valuable in connexion with reactor design.

The subjects of the sections in the Materials Handbook are listed below:—

Functions of Reactor Materials
Aluminium and its Alloys
Beryllia
Beryllium and its Alloys
Beryllium Carbide
Bismuth
Carbides
Cements and Concretes
Graphite
Hydrides
Lithium and its Alloys
Magnesium and its Alloys
Molybdenum and its Alloys
Nickel and its Alloys
Plutonium and its Alloys
Rare Earths
Silicon Carbide
Stainless Steels
Thorium and its Alloys
Titanium and its Alloys
Tungsten
Uranium and its Alloys

Vanadium and its Alloys
Zirconium and its Alloys
High-cross-section Materials
Cobalt-base Alloys.

Within each section the data are presented in logical sequence of processing operations, i.e., extraction, recovery, melting, casting, properties, metallography. Wherever possible the information is given in the form of tables and figures. As an all-over policy, in the interests of speed of publication, it was decided to minimize documentation; authors have taken the responsibility of selecting the most reliable values from all available sources. The data contained in this section of the Handbook represent information available up to 1952.

'Reactor Handbook: Engineering'; 1074 pp. Price £5. This volume deals with the following subjects:—

Light- and Heavy-Water-Cooled Systems
Liquid-Metal-Cooled Systems
Gas-Cooled Systems
Aqueous Fuel Systems
Liquid-Metal-Fuel Systems
Fused-Salt Systems
Handling and Control
Reactor Designs.

The data include, *inter alia*, the behaviour of nickel, nickel alloys, and stainless steels in contact with high-temperature water and with various hydroxide compounds employed in reactor systems.

Analysis of Thin Films

T. N. RHODIN: 'Chemical Analysis of Thin Films by X-ray Emission Spectrography.'
Analytical Chemistry, 1955, vol. 27, Dec., pp. 1857–61.

In spite of the very small fluorescent X-radiation excited in microgram samples of metals, alloys and other compounds, the sensitivity and relative simplicity of this method recommend it for metallurgical research purposes. In the experiments now reported analytical determinations were made on films of pure metals and of austenitic stainless steels deposited by evaporation on Mylar polyester substrates. Oxide films isolated from the stainless steels were also studied. Particular attention was given to clarification of the influence of the highly dispersed nature of the samples (1·0–100 γ per sq. cm.), on deviations from absorption and secondary-emission interactions characteristic of X-ray analysis of samples of normal size. Comparison of the metal-composition data obtained by X-ray methods, for metal and oxide films 300 Å or less in thickness, gave excellent agreement with those obtained independently by microcolorimetric methods. The results lead to the conclusion that accuracies within 2 per cent. or closer can be realized by X-rays in the analysis of highly dispersed samples, without application of corrections for adsorption deviations.

Descaling of Steels and Alloys after Exposure to High-Temperature Water

See abstract on p. 51.

Determination of Metals in Oils and Petroleum Products

Increasing realization of the significance of trace elements in petroleum products has recently given rise to much interest in methods for estimation of nickel, vanadium and other metals in charging stocks and residual fuels. The papers abstracted below are among the latest contributions to the subject.

E. N. DAVIS and B. C. HOECK: 'X-Ray Spectrographic Method for the Determination of Vanadium and Nickel in Residual Fuels and Charging Stocks.'
Analytical Chemistry, 1955, vol. 27, Dec., pp. 1880–4.

Attention is directed to papers recording the principal methods which have been proposed for determination of metals in oil products, and an account is given of an X-ray spectrographic procedure which has the advantages of speed, small size of sample, and accuracy comparable with that given by other methods.

The procedure recommended involves ashing of the oil sample by means of sulphuric acid, and subsequent solution of the ash. An aliquot of the ash solution is evaporated on a glass disc and is analysed, with the X-ray spectrograph, for vanadium, nickel and iron. If the iron concentration is more than ten times that of the nickel another aliquot is extracted, to remove interfering iron, and is then evaporated on a second glass disc and analysed for nickel. Apparatus for rotating the sample improves the precision of the determination.

J. T. HORECZY, B. N. HILL, A. E. WALTERS, H. G. SCHUTZE and W. H. BONNER: 'Determination of Trace Metals in Oils', *ibid.*, pp. 1899–903.

It has been shown in the literature that some of the trace metals liable to be found in oils are present as organic complexes which are volatile, and that a wet-ash procedure recovers a higher proportion of the metals than is attainable by dry ashing. It has been noted that porphyrin compounds and their metallic complexes may be present in crude oils, but no conclusive investigation has previously been reported of the applicability of wet ashing in such cases. The present authors have studied this aspect: they record the results of wet- and dry-ash procedures, tested on synthetic blends of nickel, vanadium, iron and copper derivatives of porphyrins. It is shown that all the metals mentioned are quantitatively recovered from such compounds by the wet-ash procedure, but not by the dry-ash method. The difference was less in the case of vanadium than with the other three metals. Further experiments, on recovery of nickel, iron and vanadium from natural petroleum stocks, confirmed that wet ashing is to be preferred.

J. E. MCEVOY, T. H. MILLIKEN and A. L. JULIARD: 'Spectrographic Determination of Nickel and Vanadium in Petroleum Products by Catalytic Ashing', *ibid.*, pp. 1869–72.

The experiments reported in this paper show that

trace amounts of nickel and vanadium (0·1-1000 p.p.m.), in various types of oil, can be rapidly determined, with a reasonable degree of accuracy, by ashing the oil with 7-50 weight per cent. of silica-alumina (which decomposes the sample catalytically), and by analysing the ignited product spectrographically. Nickel and vanadium sorbed on the catalyst by this procedure have the same spectro-emission characteristics as artificial standards prepared by impregnation of the same type of support with dilute nickel-nitrate and ammonium-vanadate solutions. Spectrographic determination is made by blending the ignited support with a chromium-oxide/graphite mixture, pelleting the composite, and sparking under controlled conditions.

The method has the advantage that for concentrations of about 10 p.p.m. only 2-5 grams of sample are required. The ashing stage takes approximately three hours, and the spectrographic estimation of the two elements an additional hour and a quarter.

M. WHISMAN and B. H. ECCLESTON: 'Flame Spectra of Twenty Metals Using a Recording Flame Spectrophotometer', *ibid.*, pp. 1861-9.

In connexion with the call for determination of metals in trace quantities in Diesel fuels, the U.S. Bureau of Mines has developed a flame spectrographic method, details of which are given. Spectrograms of twenty metals are shown in the paper, together with tables giving the principal flame lines and an estimated detection limit for each of the metals. Data relating to nickel are included.

Measurement of Thermal Conductivity

See abstract on p. 30.

Growth of 'Whiskers' from Metal Salts

See abstract on p. 32.

NICKEL

Nickel: Supply and Demand in the U.S.A.

U.S. DEPT. OF COMMERCE: 'A Review of Nickel in the U.S., 1946-1956.'

Published by *U.S. Government Printing Office, Washington* 25, *D.C.*, 1955; 41 pp. Price 30 cents.

In July 1954 the Department of Commerce, Business and Defense Services Administration, undertook a review of experience in the U.S.A. with regard to nickel. The results of this survey were published in a report dated November 1954, which included an analysis of probable defence requirements for 1955, and some estimate of the industrial position, based on information supplied by producers and consumers.

The purpose of the present report is to bring up to date the estimates then made and to make some forecast for 1956, together with a brief discussion of long-term prospects as affecting user industries. The scope of the report is shown below:—

Review of Consumption and Supply Experience

- Nickel Consumed in Stainless Steel in U.S.A.
- Nickel „ „ Low-Alloy Steel in U.S.A.
- Nickel „ „ Non-Ferrous Uses in U.S.A.
- Nickel „ „ High - Temperature and Electrical-Resistance Alloys in U.S.A.
- Nickel Consumed in Electroplating in U.S.A.
- Total Nickel Consumption in U.S.A.
- Nickel Stocks in Consumers' Plants.
- Total Free World Nickel Supply and U.S. Share, 1948-56.

Nickel Distribution, Defence and Non-Defence.

Outlook: Immediate Future and Long-term Prospects.

Competitive Position of Nickel, Aluminium and Copper.

Measurement of Thermal Conductivity of Nickel

P. O. DAVEY and G. C. DANIELSON: 'Thermal Conductivity of Nickel.'

U.S. Atomic Energy Commission, Ames Laboratory, Iowa State College, *Report* ISC-518, June, 1954; 24 pp.

This report opens by a discussion of the importance of a knowledge of the thermal conductivities of metals at high temperatures, and of the methods which have been proposed for determining such values. A detailed description is then given of a new type of apparatus suitable for measurements over the range 100°-1000°C. The method is based on comparison of the thermal conductivity of the test material with that of Armco iron, chosen as standard because its conductivity is known within close limits. Comparison is made by measuring the axial temperature gradient in each of three cylindrical bars in series, fastened together so that the metal of unknown conductivity is between two Armco iron standards. A heater at one end of the compound bar maintains the temperature gradient. A guard tube is used to eliminate radial heat loss. The conductivities of the sample and the standard are then inversely proportional to the measured temperature gradients. Data obtained by this method on nickel are reported for the range 150°-640°C.

Slip Patterns in Deformed Nickel and Nickel-Iron Alloys

See abstract on p. 39.

Inter-Diffusion of Copper and Nickel

See abstract on p. 36.

Sorption of Carbon Monoxide on Nickel

Z. ODA: 'Sorption of Gases on Evaporated Nickel Films. II. and III. Adsorption and Decomposition of Carbon Monoxide at Low Pressures.'

Bull. Chemical Soc., Japan, 1955, vol. 28, pp. 281-4, 285-90. Abstract in *Jnl. Applied Chemistry*, 1955, vol. 5, Dec., ii, p. 776.

Adsorption of Hydrogen and Carbon Monoxide by Nickel

(1) M. McD. BAKER, G. I. JENKINS and E. K. RIDEAL: 'The Adsorption of Hydrogen by Nickel.'

Trans. Faraday Soc., 1955, vol. 51, Nov., pp. 1592–6.

Study of the interaction of hydrogen with nickel films. It is shown that in addition to adsorption, a further amount of gas is taken up and that the uptake involves an energy of activation. The authors present evidence refuting the view that lattice solution is involved: the existence of lattice vacancies or spaces between macrocrystals as sites for the further uptake is suggested.

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(2) M. McD. BAKER, G. I. JENKINS and E. K. RIDEAL: 'The Adsorption of Carbon Monoxide by Nickel', *ibid.*, pp. 1597–601.

The chemisorption of carbon monoxide is one of the fundamental steps involved in its varied catalytic reactions. In this paper the adsorption of carbon monoxide by evaporated nickel films is investigated at various temperatures.

Diffusion of Hydrogen in Nickel

M. L. HILL and E. W. JOHNSON: 'The Diffusivity of Hydrogen in Nickel.'

Acta Metallurgica, 1955, vol. 3, Nov., pp. 566–71.

Diffusivity of hydrogen in nickel was determined at 380°–1000°C. from non-steady rates of hydrogen evolution from 0·3–0·5 in. diameter cylinders. Initial charging of the specimens was effected by two methods: (1) 'thermal' charging with up to 16·7 ml./100 g. hydrogen by saturating for 15 hours at 1 atmosphere and 1150°–1400°C.; (2) annealing at 1000°C. for two hours and then 'pressure' charging with up to 48 ml./100 g. hydrogen by saturating for 18 hours at 600°C. under pressures up to 67 atmospheres. Evolution rates into a vacuum were measured (using apparatus which is described and illustrated) by (*a*) rate of pressure increase in a known collecting volume and (*b*) pressure drop across a constriction in the gas-transfer line.

From the results obtained it is concluded that the evolution of hydrogen from nickel is a diffusion-controlled process, obeying conventional diffusion kinetics in the temperature range used in these experiments. No perceptible variation in the diffusion coefficient with concentration was found in the range 0–50 ml./100 g., nor was there any evidence that the diffusivity varies with thermal history in the manner previously found in iron. Observations made are in good accord with the hypothesis that the hydrogen is situated predominantly within, and diffuses among, the octahedral interstices of the nickel lattice. The experimentally determined entropy of activation is negative, suggesting that the diffusion is accompanied by little, if any, lattice distortion.

Descaling of Metals after Exposure to High-Temperature Water

See abstract on p. 51.

Joining of Nickel and Nickel Alloys

See abstract on p. 39.

Reactor Materials and Design

See abstract on p. 28.

Nickel Catalysts: Thermomagnetic Analysis

J. A. SABATKA and P. W. SELWOOD: 'Thermomagnetic Analysis of Catalytically Active Nickel.'

Jnl. Amer. Chemical Soc., 1955, vol. 77, Nov. 20, pp. 5799–802.

This paper describes an improved form of apparatus (for thermomagnetic analysis) which had been previously referred to in this journal. Experiments made with it show that massive and disperse nickel have the same magnetic moment at 0°K. (−273°C.). The method was used also for study of several commercial nickel catalysts.

Gravimetric Determination of Nickel

C. V. BANKS and D. T. HOOKER: '4-Methyl- and 4-Isopropyl-1,2-Cyclohexanedionedioxime. Gravimetric Reagents for Nickel and Palladium.'

Analytical Chemistry, 1956, vol. 28, Jan., pp. 79–81.

The desirability and applicability of water-soluble *vic*-dioximes as analytical reagents have been urged in the literature, and it has been pointed out that compounds with oxime groups attached to the adjacent carbon atoms of six- or seven-membered alicyclic rings possess several advantages over other *vic*-dioximes. They have a much higher molar solubility in water than have straight-chain aliphatic *vic*-dioximes of similar or even lower molecular weights, and other advantages are that they are sensitive for the detection of nickel and palladium in sub-microgram amounts, that they quantitatively precipitate these metal ions from acid solutions, and that they have high equivalent weights.

The authors review the limitations of 1,2-cyclohexanedionedioxime (nioxime), of 1,2 cycloheptanedionedioxime (heptoxime), and of alicyclic *vic*-dioximes with less than six or more than seven carbon atoms in the ring. In view of the information thus summarized, study of substituted alicyclic *vic*-dioximes containing a six-membered ring appeared most promising. A series of seven alkyl-1, 2-cyclohexanedionedioximes (alkylnioximes) was investigated. Only 4-methylnioxime and 4-isopropylnioxime appeared likely to yield useful results. This paper reports closer investigation of these two compounds.

4-Methylnioxime was found to be an excellent reagent for the gravimetric determination of both nickel and palladium. In the determination of nickel this reagent has more favourable attributes than any *vic*-dioxime suggested to date. It is water-soluble (3·4 grams per litre at 25°C.), quantitatively precipitates nickel as a scarlet-coloured complex down to pH 3, forms a precipitate free from contamination of excess reagent, which filters easily and does not tend to creep, and has a high equivalent weight. The only *vic*-dioxime known to possess equivalent

virtues is heptoxime, but the cost of preparing this reagent is much greater than for 4-methylnioxime. 4-Methylnioxime is equally useful for the gravimetric determination of palladium.

4-Isopropylnioxime, while being less soluble (0·75 gram per litre at 25°C.), is still sufficiently soluble to be a very useful reagent for the gravimetric determination of small amounts of nickel and palladium.

Full details of procedures are given for the estimation of both metals.

Determination of Metals in Oils and Petroleum Products

See abstracts on pp. 29–30.

Exchange Reactions of Metal Oxides

E. R. S. WINTER: 'Exchange Reactions of Solid Oxides. Part VI. The Reactions of Carbon Monoxide, Carbon Dioxide, and Oxygen on Cuprous Oxide, Nickel Oxide, and Chromium Oxide.'

Jnl. Chemical Soc., 1955, Aug., pp. 2726–40.

The reaction $2CO+O_2 \rightarrow 2CO_2$ was studied, by using mainly the stoicheiometric mixture of reactants, on NiO, Cr_2O_3 and Cu_2O, at pressures of 1–9 cm. and temperatures from 10° to 155°C.

Extraction of Trace Amounts of Nickel from Cobalt Salts

R. A. SHARP and G. WILKINSON: 'Solvent Extraction Separation of Cobalt and Nickel with Thiocyanate and the Preparation of Nickel-free Cobalt Salts.'

Jnl. Amer. Chemical Soc., 1955, vol. 77, Dec. 20, pp. 6519–21.

Report of work done in connexion with the requirement for very pure cobalt metal foil targets for cyclotron bombardment.

The optimum procedure was found to be use of cobalt nitrate as the initial material, extraction of a solution 0·35 *M* in cobalt and 5 *M* in NH_4SCN, with hexone at 1°, washing this once with 5 *M* NH_4SCN at 1°, and evaporation of the organic phase to dryness over H_2SO_4. With quantitative recovery of the organic phases, this procedure is found to yield more than 99·5 per cent. of the original cobalt, with the nickel impurity reduced by a factor of 10^4. Recycling will give even higher purifications.

Toxicity of Nickel Carbonyl

I. V. SANOTSKII: 'Action Mechanism of Nickel Carbonyl.'

Farmakol. i Toksikol., 1955, vol. 18, No. 2, pp. 48–50.
Chemical Abstracts, 1955, vol. 49, Sept. 25, p. 12,732.

Experiments on mice showed that nickel carbonyl (0·01 mg./L.) caused a noticeable decrease in oxygen consumption, and that at 0·2 mg./L. there was a very sharp drop. These and other observations suggest that the effect is one of paralysis of pleural reflexes, and it is suggested that this finding indicates methods for developing more effective antidotes.

Growth of 'Whiskers' from Metal Salts

S. S. BRENNER: 'The Growth of Whiskers by the Reduction of Metal Salts.'

Acta Metallurgica, 1956, vol. 4, Jan., pp. 62–74.

The experimental work in this paper is prefaced by a useful review of the literature on production of metals in the form of fine filaments or whiskers, by reduction of their salts. The original work reported is concerned with growth of whiskers by reduction of the halides of iron, copper, silver, nickel, cobalt, platinum and gold. Detailed study (made chiefly on copper) demonstrates that the growth of whiskers proceeds at the tip and that material is furnished by condensation of the freshly formed metal vapour.

See also—

E. E. THOMAS: 'Tin Whisker Studies. Observation of Some Hollow Whiskers and Some Sharply Irregular External Forms', *ibid.*, p. 94.

ELECTRODEPOSITION AND OTHER COATING METHODS

Documentation on Deposition of Metals and Related Subjects

N. HALL: 'Technical Developments of 1955.'

Metal Finishing, 1956, vol. 54, Jan., pp. 43–52.

Review, supported by 285 references to the literature of 1955, classified in sections relating to cleaning; pickling; polishing; plating and anodizing of aluminium; deposition of nickel, chromium, copper and other metals, and of alloys; metallizing; conversion films; testing and control methods; miscellaneous subjects such as specifications, equipment, oxide coatings, etc.

Automatic Nickel/Chromium Plating of Automobile Components

'A Description of the Automatic Nickel/Chromium Plating Line in Operation at the Luton Works of Vauxhall Motors, Ltd.'

Metal Finishing Jnl., 1956, vol. 2, Jan., pp. 5–12, 22.

The steady increase in the output of motor cars during recent years, and the incorporation, in their design, of substantial areas of nickel/chromium plating, have led to considerable extension of electroplating facilities at the works of car manufacturers. Where possible, in the interests of efficiency, speed, and economy, the trend has been towards introduction of semi- or fully automatic plant. The first fully mechanized plating installation in a British motor-car plant has now been in operation for more than a year in the Luton works of Vauxhall Motors, Ltd., where it is used for plating of sheet-metal pressings. Overall dimensions are 180 ft. long × 30 ft. wide and the whole plant comprises 22 pairs of process tanks, arranged in two parallel rows, each tank being 6 ft. wide and 7 ft. deep. The principal feature of the set-up is the process conveyor system,

which carries the suspended loads of components automatically through a sequence of 22 operations from cleaning to final rinsing after chromium plating.

Prior to introduction of the work into the automatic plant, all components are manually copper plated from a solution containing additions which will ensure a semi-bright and 'levelled' deposit. The coating so deposited is of a minimum thickness of 0·001 in., and the work is subsequently polished, to ensure a smooth surface, on which the copper/nickel/chromium coating is electrodeposited in the automatic plant.

This article is well illustrated by photographs of various sections of the plant, and by a diagram of the layout of the plating area, showing the disposition of the automatic installation. Some details of construction and operation of the plant are also given, e.g., relating to tank construction, heating of solutions, filtration, current, agitation, etc. Some notes are added on 'teething troubles' initially encountered and means which have been adopted to overcome them.

Bright Nickel-Plating Process of High Levelling Capacity

'Bright Nickel Plating.'

Metal Industry, 1956, vol. 88, Jan. 27, pp. 71–2.
See also *Electroplating*, 1956, vol. 9, Feb., p. 53.

The article gives details of Efco-Udylite bright nickel-plating process No. 514, which has recently been introduced into the United Kingdom by Electro-Chemical Engineering Company, Limited. The principal advantage claimed for this process is that the solution has much improved filling or levelling properties and that it is characterized by an unusually wide bright-plating range.

The electrolyte used contains (g./L.) nickel sulphate ($NiSO_4.6H_2O$) 300; nickel chloride ($NiCl_2.6H_2O$) 60; boric acid (H_3BO_3) 40, with specified amounts of three proprietary brightening agents and an anti-pitting medium. The functions of the respective components of the solution are discussed and operating conditions are detailed.

A variant of the 514 process, designated 514H, is specially recommended for plating on copper-plated zinc-base die castings, and is also particularly suitable as a general-purpose solution in automatic plants, where its high cathode current density permits relatively rapid specification plating. The composition of the 514H solution is given as (g./L.) nickel sulphate ($NiSO_4.6H_2O$) 60; nickel chloride ($NiCl_2.6H_2O$) 225; boric acid (H_3BO_3) 40. The same types of brightening agents are used, but in slightly different proportions from those employed in the 514 bath. The non-pitting agent is identical in both cases. It is noted that this solution is capable of withstanding a contamination of approximately nine times the amount of zinc which can be tolerated by the standard 514 solution: a maximum tolerance is obtained with a nickel-sulphate concentration between 44 and 60 g./L. When contamination with zinc is not a factor of prime importance (e.g., when plating steel) the nickel sulphate may be higher than 60 g./L.

Structure of Electrodeposited Nickel

B. C. BANERJEE and A. GOSWARMI: 'Electron-Diffraction Study of the Structure of Electrodeposited Nickel.' *Jnl. Scientific and Industrial Research, India*, 1955, vol. 14, B, pp. 322–4.

The structure and orientation of electrodeposited nickel were studied on deposits made under the following conditions:

Bath composition (g./L.): nickel sulphate 280, boric acid 31; pH 1·0–5·9; temperature 26° and 75°C.; current density 10 and 70 milliamp./cm². The deposits were made on plane surfaces of mechanically polished 70–30 brass.

The observations made are summarized in *Journal of Applied Chemistry*, 1955, vol. 5, Dec., ii, p. 724.

Resistance of Nickel/Rhodium-Plated Parts to Moist Ozone

F. J. BIONDI: 'Corrosion-Proofing Electronic Parts against Ozone.'

Ceramic Age, 1955, vol. 66, Oct., p. 39.

The interaction of silent electric discharge with the oxygen in the air produces ozone which, in addition to being toxic and degrading to organic insulators, also corrodes certain metals. The conditions which generate ozone usually also produce oxides of nitrogen, which, in the moist ambient atmosphere, add to the corrosion problem. In some cases conditions conducive to generation of ozone can be avoided by suitable design, but in others this is impossible, and the atmosphere of operation is inevitably beyond the control of the designer. Military electronic gear is particularly likely to work under high-temperature moist conditions which are unfavourable to efficiency.

This short note reports experience in the Bell Telephone Laboratories, to which, during 1953, a number of magnetrons were returned from the field with reports of open heaters. Examination showed that the heaters were not actually open, but appeared to be so due to the presence of high-resistance corrosion films on the contacts. Examination of methods by which corrosion-proofing could be effected has led to development of the following plating sequence:—Electrodeposition of nickel (0·7 mil. thickness); sintering the plated part, in hydrogen, for 15 minutes at 925°C.; electrodeposition of a nickel-strike coating, and finishing with a 0·025-mil. coating of rhodium. Specimens so treated easily withstand the conditions involved in processing of magnetrons, and exposure to hot moist ozone produces no attack of the surface. Ferrous parts of magnetrons are now being regularly treated by this procedure, and are remaining free from corrosion in service.

Specifications Relating to Electrodeposited Coatings

AMER. SOC. TESTING MATERIALS: 'Specifications and Tests for Electrodeposited Metallic Coatings.' Sponsored by American Society for Testing Materials and American Electroplaters' Society.

Published by *American Society for Testing Materials, Philadelphia, Pa.*, Sept., 1955; 92 pp. Price $1.40.

The brochure has been compiled to bring together

all the A.S.T.M. specifications and methods of test relating to electrodeposited metallic coatings. It contains the most recently revised forms of the following schedules:—

A.164–55 Specification for Electrodeposited Coatings of Zinc on Steel.

A.165–55 Specification for Electrodeposited Coatings of Cadmium on Steel.

A.166–55T Specification for Electrodeposited Coatings of Nickel and Chromium on Steel.

A.219–54 Methods of Test for Local Thickness of Electrodeposited Coatings.

B.117–54T Method of Salt-Spray (Fog) Testing.

B.141–55 Specification for Electrodeposited Coatings of Nickel and Chromium on Copper and Copper-base Alloys.

B.142–55 Specification for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-base Alloys.

B.177–55 Recommended Practice for Chromium Plating on Steel for Engineering Use.

B.183–49 Recommended Practice for Preparation of Low-Carbon Steel for Electroplating.

B.200–55T Specification for Electrodeposited Coatings of Lead on Steel.

B.201–55T Specification for Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die-Cast Surfaces.

B.242–54 Recommended Practice for Preparation of High-Carbon Steel for Electroplating.

B.252–53 Recommended Practice for Preparation of Zinc-base Die Castings for Electroplating.

B.253–53 Recommended Practice for Preparation of, and Electroplating on, Aluminium Alloys.

B.254–53 Recommended Practice for Preparation of, and Electroplating on, Stainless Steel.

B.281–53T Recommended Practice for Preparation of Copper and Copper-base Alloys for Electroplating.

B.287–54T Method of Acetic Acid-Salt Spray (Fog) Testing.

Production of Diffused Nickel-Cadmium Coatings

R. W. MOELLER and W. A. SNELL: 'Diffused Nickel-Cadmium as a Corrosion Preventive Plate for Jet Engine Parts.'

Plating, 1955, vol. 42, Dec., pp. 1537–40.

This paper, presented at the 1955 meeting of the American Electroplaters' Society, describes research carried out in developing the composite coating referred to in a paper by NOBLE and SHARP (see abstract in *Nickel Bulletin*, 1955, vol. 28, No. 12, pp. 225–7).

The test programme had been designed to cover the requirement of a coating which, at a thickness of 0·0003–0·0005 in., would give adequate protection at temperatures up to 1000°F. (540°C.). Zinc, cadmium or tin alone was eliminated for use at such temperatures, due to the tendency of these metals to alloy with the steel and cause stress-corrosion cracking; chromium plating in the thickness required gave inadequate protection, and was considered unsuitable for coating where uniform plating of complex shapes was called for: electrodeposited nickel, in the thickness range specified, failed to meet the requirements laid down for corrosion-resistance. In early tests, electroless nickel did not meet the corrosion specification, and although recent tests have shown more satisfactory behaviour in that respect, the tendency of hardened base material so coated to show a fall in fatigue strength has retarded the acceptance of electroless nickel as a component of the diffused coatings. A nickel-zinc coating gave generally good protection against corrosion, but was somewhat inferior to nickel-cadmium.

Study of ratios of plate thickness of the two metals, made to determine optimum corrosion-resistance consistent with freedom from stress-alloying, established that the nickel coating should be 0·00005 in. or more, and that coatings consisting of 0·0003 in. nickel+0·0001 in. of cadmium, diffused at 630°F. (332°C.) for one hour, would withstand 100 hours of salt spray without rusting, even if the material had been heated for 23 hours at 700°F. (370°C.) and one hour at 1000°F. (540°C.) prior to corrosion test.

Successful use of the process is dependent on a number of factors, some of which are briefly noted below.

Low-stress deposits are desirable, and, it has been found desirable to use chloride or sulphate-chloride baths containing no organic addition agents. Coatings produced from the sulphamate type of bath (without the additives necessary to produce compressive stress) are also showing promise. Cadmium coatings deposited from the cyanide bath have proved satisfactory and, in order to prevent a stained and fingerprinted appearance, the cadmium plating is treated, before diffusion heat-treatment, by immersion in chromate conversion solutions.

In plating intricately shaped parts a relatively elaborate system of conforming anodes and thieves is necessary: typical fixtures used for plating various jet-engine parts are shown in this paper.

Since it is imperative that no free cadmium be available on engine parts which operate under stress at temperatures above the melting point of cadmium, it is strongly recommended that the plater observe the following precautions:—

(1) Regulate the process of nickel deposition to secure a satisfactory deposit of at least 0·00005 in. nickel in all areas where nickel-cadmium plate is required.

(2) Deposit cadmium only on areas which have been previously nickel plated.

(3) Diffuse all parts at 630°F. (332°C.) for one hour prior to engine use.

(4) Prevent transfer of cadmium from plating hooks, fixtures, etc.

(5) Reject plate which shows 'balled' cadmium, indicating inadequate diffusion.

Electroless Nickel Plating: German Experiments

–. BOSDORF: 'Electroless Nickel Plating and its Applications.'

Electroplating, 1955, vol. 8, Dec., pp. 415–17, 428.

The author reports experiments made in the laboratories of Langbein-Pfanhauser Werke, A.G., using acid and alkaline solutions basically similar to those developed by Brenner and Riddell, but modified by the additions shown in the table below:—

	Acid Solution g./L.	*Alkaline Solution* g./L.
Nickel chloride ..	30	30
Sodium hypophosphite	10	10
Ammonium chloride	—	50
Sodium citrate ..	—	100
Salt of glycollic acid	50	—
pH	5·5	10·5
Operating temperature	90°–100°C.	90°–100°C.

Curves in the paper show rate of deposition, as a function of time, for both solutions. A study of the thickness of deposit obtained in a given time indicated that this increased with increase of nickel-ion or hypophosphite concentration, up to a limiting value.

It is pointed out that control of pH is a significant factor in deposition by the electroless process. Formation of hydrogen ions during the reaction results in a fall in pH, which may lead to re-dissolution of the nickel: on the other hand, the reducing power of the hypophosphite is decreased by raising the pH. For this reason buffer compounds are added to the solution (e.g., sodium acetate in an acid bath) in order to maintain it at a pH between 4 and 6. Provided that optimum operating conditions are maintained, 0·6–1 mil. of plating can be deposited per hour.

The levelling of the electroless coatings, and the possibility of filling cracks in the basis material, are illustrated by photographs of cross sections of an electroless nickel coating deposited on a cast bronze water tap.

Electroless Nickel Plating of Aluminium

T. A. DICKINSON: 'A Brief Note on the Electroless Deposition of Nickel on Aluminium in an American Works.'

Metal Finishing, 1955, vol. 1, No. 12, Dec., pp. 528–537.

This note refers to the successful use, by Northrop Aircraft, Inc., of an electroless nickel-plating process for coating many of the aluminium parts used in the Scorpion F-89D aircraft and the Snark SM-62 strategic missiles now in production at Hawthorne, California. Coatings of this type are in many cases replacing anodic finishes, giving economic advantages, and providing a surface which can be soldered or brazed. The plating solution used is a proprietary one, developed by Allied Research Products, Inc., Baltimore, Md., U.S.A.

Large stainless-steel wire baskets and specially designed racks are used to provide good drainage, and to hold the work in such a way that the surfaces to be plated will be exposed in the most favourable way to the solution. The operating cycle comprises cleaning in an air-agitated detergent solution for at least five minutes at 180°F. (83°C.), hot-water rinsing, dipping in a deoxidizing solution (at room temperature), cold-water spray rinsing, and immersion in the plating solution for about three minutes. Finishing comprises spray rinsing and drying: parts which are of shapes preventing easy drainage are oven-dried after final rinsing. It is reported that some difficulty has been experienced in processing parts carrying stains or other discolourations resulting from heat-treatment, but this has been overcome by etching any marked parts before cleaning. It is stated by Northrop that savings resulting from elimination of operations required for anodized parts will, in one year, more than offset the cost of the new electroless nickel-plating installation.

Metal Spraying: Recommended Procedure

'Metal Spraying Flat Surfaces. A New American Code of Recommended Practice.'

Electroplating, 1956, vol. 9, Jan., pp. 19–22.

This article summarizes the contents of a publication recently issued by the American Welding Society under the title 'Metallizing Flat Surfaces.' It refers specifically to the application of sprayed coatings to lathe and other machine-tool ways, gibs, machine-tool beds, mis-machined components, platens and similar surfaces. As the second in a series dealing with metal spraying for building up, this publication must be considered in conjunction with the A.W.S. Recommended Practice for metal spraying of shafts and other cylindrical parts.

The procedure detailed in the present publication includes surface preparation, means to be adopted to ensure surface bonding, and metal-spraying technique. Causes of cracking or lifting at the corners or edges of the sprayed deposit are discussed, and methods of avoiding such defects are suggested. Finishing procedure is also recommended.

The mechanical and physical properties typical of sprayed coatings of various metals are listed, including reference to coatings of nickel, Monel and stainless steel. The information on U.S. methods is supplemented by brief reference to British practice in spraying flat surfaces, in so far as the technique differs in the two countries.

Nickel-Plated Steel Sheet and Plate: 'Lectroclad'

L. L. WILLIAMS: 'Nickel-Plated Steel Sheet and Plate.'

Materials and Methods, 1955, vol. 42, Nov., pp. 112–14.

This article describes the properties and uses of sheet or plate coated, on one or both sides, with nickel electrodeposited by a proprietary process developed by Bart Manufacturing Corporation. The application of such material is for components in which corrosion protection is required, but for

which steel clad with nickel (or with other corrosion-resisting materials), in heavier thickness, would not be economic and/or suitable.

The plating is produced (from a Watts-type bath of high metal content) to the following specification: nickel 99·00–99·95, iron 0·1 max., copper 0·02 max., manganese 0·010 max., lead 0·005 max., cobalt 0·10 max., per cent.). It is specified that the tensile properties of the coating shall be equal or superior to those laid down for the basis metal, and that ductility shall be such that no flaking, checking or spalling shall occur in a specified bend test, with the plate in tension or in compression. As a result of special etching of the base metal before plating, and of strict control of plating operations, bond strength of the coating to the base is normally equal to the yield strength of the base metal. A further test requires that no blistering shall occur on heating a series of ½-in. areas to 1000°–1100°F. (540°–595°C.).

Corrosion testing may be done by one of two methods:

(*a*) Immersion in aerated water at 185°±15°F. (85°±8°C.) for 30 minutes. On removal from the bath the plate must show no brownish stains, and it is again examined after a further 15–30 minutes.

(*b*) The surface is cleaned with 10 per cent. hydrochloric acid and water-rinsed, and a mixture made from a 10 per cent. copper sulphate and a 10 per cent. hydrochloric acid solution is applied to it. Under this test it is required that there shall be no sign of copper plating out (and resulting in copper-coloured spots), which would occur in any areas in which the solution came into contact with unplated ferrous material.

With regard to fabrication of the coated sheet and plate, the following notes are added:

Since the nickel coating is adherent, ductile, and strong, fabrication characteristics of the composite depend mainly on those of the basis steel. No special equipment is needed, but certain techniques (similar to those used for clad plates) are desirable in order to protect the nickel-plated surface from damage during fabrication. During storage and transportation the plated sides are faced to each other and separated by wood strips, or covered with plastic coating which can subsequently be stripped off.

In flame cutting no pre-heating is required, and the material can be flame cut and bevelled for welding at speeds normally used for carbon-steel plates of equivalent thickness. In forming operations the working surface of equipment used must be smooth, and its surface, and that of the nickel plating, must be free from chips or other foreign matter. In addition, a piece of heavy kraft paper should be inserted between the plated surface and the die or roll. Soapstone is the only marking material recommended for use on the nickel surface.

Either metal-arc or inert-gas arc welding is recommended for the plated material: for the former process INCO Rod No. 141 with a current of about 100 amp. (for ⅛-in. rod), is preferred, and when the inert-gas arc-welding procedure is employed INCO Wire No. 61 should be used, with a current of about 200 amp., voltage about 24., and welding speed about 20 in. per minute (for 0·045-in. wire.)

In cleaning, shop dirt, greases and oils can be removed by a standard alkaline cleaner, and for stains a phosphoric-acid solution may be used. Residual stains may be eliminated by swabbing the surface with a mixed solution of sulphuric and chromic acids, but the cleaner must be thoroughly rinsed off with cold water before the work is dried. The material may also be cleaned by sand blasting for a limited period, or by wire brushing, using a stainless steel or nickel brush.

Effects of Metal-Finishing Effluents on Sewage Purification

A. E. J. PETTET: 'Effect of Metal Finishing Wastes on Sewage Purification.'

Bull. Inst. Metal Finishing, 1955–56, vol. 5, Winter, pp. 225–52.

Discharges of waste waters from nickel-plating and other metal-finishing plants fall into two categories: (*a*) the continuing discharge of considerable quantities of dilute rinsing and quenching waters, and (*b*) the batch discharge of smaller amounts of concentrated spent pickling, anodizing, and cleaning solutions, and, occasionally, of spoilt plating solutions. Typical values for concentration of plating wastes in some waters examined at the British Water Pollution Research Laboratory are shown, and the concentrations of metal and salts liable to be present in standard plating solutions are also quoted.

The essential features of the combined physical and biological processes by which sewage is treated are reviewed as a background to discussion of the extent to which metal- finishing contaminant materials can act as interferants, particularly in the biological stages. The subject is then discussed in sections relating to the individual stages of sewage collection and treatment, considering first the effects of effluents on and in sewers, and subsequently their influence in the respective treatment processes.

Review of existing data, together with the results of detailed tests now reported, makes possible a number of practical conclusions with regard to permissible concentrations of metals and ions liable to occur in wastes from plating solutions and other liquids used in metal-finishing plants. The information presented indicates that, in order to avoid serious interference with treatment of sludge, chemical pre-treatment of such wastes before discharge to the sewer is often necessary. Details of some suitable methods are given.

NON-FERROUS ALLOYS

Inter-Diffusion of Copper and Nickel

M. L. MEHTA and H. J. AXON: 'The Influence of Thin Intermediate Layers on Interdiffusion in Copper-Nickel Couples.'

Acta Metallurgica, 1955, vol. 3, Nov., pp. 538–41.

Although much attention has been devoted to the

study of interdiffusion of copper and nickel (to the major literature of which the authors give reference), no published information could be traced on the effect of an intermediate layer of a third metal on diffusion in a copper-nickel couple. Such effects were therefore studied, using thin (0·00002 in.) electrodeposited interlayers of gold, tin, zinc and cadmium between fully annealed nickel rod and electrodeposited copper.

The composite specimen and a control specimen having no interlays were slowly heated from room temperature to the diffusion annealing temperature (950°C.), held for 140 hours at that temperature, and allowed to cool in the furnace.

The results are shown in a series of curves recording (*a*) concentration-penetration, and (*b*) variation of diffusion coefficient with concentration for the respective composites. The observations made lead to the conclusion that gold or zinc has little effect on the chemical diffusion coefficients of the copper-nickel system, although a very slight retardation was detected in the range 60–80 at. per cent. copper. Tin behaves differently from cadmium, in that it accelerates diffusion of nickel into copper but retards that of copper into nickel, whereas cadmium has an accelerating effect in both cases.

Copper-Nickel-Chromium Alloys: Constitution

J. L. MEIJERING, G. W. RATHENAU, M. G. VAN DER STEEG and P. B. BRAUN: 'A Miscibility Gap in the Face-Centred Cubic Phase of the Copper-Nickel-Chromium System.'

Jnl. Inst. Metals, 1955–56, vol. 84, Jan., pp. 118–20.

The authors studied the copper-nickel-chromium system by metallographic and X-ray diffraction methods, giving particular attention to the isothermal section at 930°C. In addition to the body-centred cubic phase rich in chromium, two face-centred cubic phases were found, although the binary copper-nickel alloys show complete miscibility.

Nickel-Rhenium Alloys

S. A. POGODIN and M. A. SKRYABINA: 'Investigation of the System Nickel-Rhenium.'

Izvest. Sektora Fiz.-Khim. Anal., Inst. Obshchei i. Neorg. Khim., Akad. Nauk S.S.S.R., 1954, vol. 25, pp. 81–8.

The system was studied from 0 to 100 per cent. rhenium, by thermal analysis, microstructural examination and X-ray investigation. The observations made are summarized in *Chemical Abstracts*, 1955, vol. 49, p. 14,624.

Nickel-Tellurium Alloys

V. P. ZHUZE and A. R. REGEL: 'Electrical Properties of Alloys of the System NiTe-$NiTe_2$.'

Zhur. Tekhn. Fiziki, 1955, vol. 25, pp. 978–83.
Metallurgical Abstracts, 1955, vol. 23, Dec., p. 292.

Electrical conductivity, and its temperature coefficient, thermal e.m.f. and thermal conductivity, were studied on nickel-tellurium alloys containing 10–40 per cent. nickel. Both cast and powder-produced specimens were examined. The powder-metallurgy samples showed approximately twice the conductivity of the cast specimens, but the ratio varied from one composition to another. The results show a sharp maximum of conductivity and thermal e.m.f. corresponding to the compounds $NiTe_2$ and NiTe.

Nickel-Zirconium Alloys

S. A. POGODIN and V. I. SKOROBOGATOVA: 'Alloys of Nickel and Zirconium.'

Izvest. Sektora Fiz.-Khim. Anal., Inst. Obshchei i. Neorg. Khim., Akad. Nauk S.S.S.R., 1954, vol. 25, pp. 80–1.

Report of study of the system from 0 to 66 per cent. zirconium, by physiochemical methods.

For results, see *Chemical Abstracts*, 1955, vol. 49, p. 14,624.

B. T. MATTHIAS and E. CORENZWIT: 'Superconductivity of Zirconium Alloys.'

Physical Rev., 1955, vol. 100, Oct. 15, pp. 626–7.

Alloys of zirconium with elements of the VIII th. Group (iron, cobalt, nickel, platinum, palladium, rhodium, ruthenium, iridium, osmium) are shown to have superconducting transition temperatures substantially higher than those characteristic of zirconium. The observations confirm the conclusion drawn from earlier work (*ibid.*, 1955, vol. 97, p. 74) that a maximum in superconducting temperatures occurs for elements, compounds and alloys which have an average valence electron per atom ratio near 5 or 7.

Anelastic Phenomena in Gold-Nickel Alloys

C.-Y. ANG, J. SIVERTSEN and C. WERT: 'Some Anelastic Phenomena in Alloys of Gold and Nickel.'

Acta Metallurgica, 1955, vol. 3, Nov., pp. 558–65.

It has been noted that certain substitutional alloys exhibit a type of anelastic behaviour which appears to be related to a change of order under stress. The authors review existing data and report observations made on gold-nickel polycrystalline alloys containing 8, 30, 40, 59, and 91 at. per cent. nickel. The results given in the paper relate mainly to the 30 per cent. nickel alloy, as being broadly typical of the behaviour of all the alloys examined.

In these alloys two relaxation peaks were found between room temperature and 600°C.: one of them was identified as the order peak, the origin of the other is uncertain. Both peaks exist in alloys quenched from the solid-solution region of the constitutional diagram. On annealing of these quenched solid-solution alloys both peaks disappear: the disappearance of the order peak is associated with formation of the mixture phases, whereas the other peak vanishes at a much earlier stage of the annealing. The kinetics of the disappearance of the order peak are consistent with measurements of the kinetics of formation of the mixture phases as determined by independent methods. Although the nature of the second peak has not yet been established, study of the influence of grain size leads the authors to conclude that neither of the points is the grain-boundary peak.

Nickel-containing Copper-Tin Bronzes

INTERNATIONAL NICKEL CO., INC.: 'Engineering Properties and Applications of NI-VEE Bronzes.' Oct., 1955; 25 pp.

Relatively small amounts of nickel, added to brasses and bronzes, have been used for many years to improve strength, pressure-tightness, toughness and other properties, and it has more recently been recognized that somewhat larger amounts of nickel further broaden the scope of usefulness of a group of bronzes which are of major importance in many industrial applications. Nickel of the order of 3–8 per cent. is found to ensure properties which are substantially higher than those obtainable from bronzes containing less nickel.

This publication forms a reference book of information on the properties of a series of bronzes containing 88–70 per cent. of copper, with about 5 per cent. each of nickel and tin, and with or without lead and/or zinc. The first section gives a detailed survey of properties and treatment: the second contains a survey of applications for which the respective grades of these materials are suitable.

See table below for typical compositions of alloys covered.

Cu %	Ni %	Sn %	Pb %	Zn %
*88	5	5	—	2
*87	5	5	1	2
80	5	5	5	5
80	5	5	10	—
70	5	5	20	—

* These bronzes meet the requirements of A.S.T.M. Specification B 292–54T.

A particularly significant feature of these bronzes is that their mechanical properties, which are already high in the as-cast condition, can be further enhanced by heat-treatment, which causes precipitation hardening. The effect begins to operate at a level of about 2 per cent. each of tin and nickel, and optimum results are obtained at the 5 per cent. tin, 5 per cent. nickel level. Characteristic mechanical properties are shown in charts and tables.

These bronzes, in the as-cast condition, can be machined at approximately the same rates as the corresponding nickel-free alloys: in the heat-treated condition the higher hardness is associated with less easy machining, and it is desirable, where possible, to machine in the as-cast condition and subsequently harden as required. The nickel-containing bronzes are characterized by a resistance to metal-to-metal seizure which makes them particularly suitable for use as bearings.

The inherently good corrosion-resistance characteristic of bronze alloys is enhanced, for certain applications, by the presence of nickel, and for conditions which involve combined erosion and corrosion these alloys, chill-cast or hardened by heat-treatment, give outstandingly good service. They are suitable for all applications in which bronzes are normally used at temperatures up to about 450°F. (230°C.): prolonged exposure at such temperatures does not significantly alter their properties, and the strengthening or hardening resulting from long exposure at elevated temperatures may be advantageously used in such applications as valves, discs, pump seals, etc.

The brochure is well illustrated by typical examples of applications for which the precipitation-hardening nickel bronzes have already established themselves: the information has been contributed by some thirty fabricators and users.

Properties of Age-Hardenable Aluminium-Nickel-Copper Alloys

J. P. DENNISON: 'Observations on the Mechanical Properties of Two Age-Hardenable Copper-Aluminium Alloys.'

Jnl. Inst. Metals, 1955–56, vol. 84, Jan., pp. 115–17.

Observations on the creep characteristics of aluminium bronzes containing cobalt or nickel have been reported in earlier papers (*ibid.*, 1953–54, vol. 82, p. 117; 1954–55, vol. 83, p. 465; *Nickel Bulletin*, 1955, vol. 28, No. 8, p. 126).

This paper deals with the effect of ageing, at 400°C., on the mechanical properties of two of the alloys used in the earlier experiments. Their composition is shown below:—

Cu %	Al %	Co %	Ni %	Remainder %
92·58	6·03	1·37	—	0·02
86·41	6·88	1·72	4·97	0·02

Graphs show (1) effect of time of precipitation treatment at 400°C.; (2) effect of cold work on the properties of solution-treated alloys; (3) effect of time of precipitation treatment on the properties of the alloys cold-worked after solution treatment. Particular attention is directed to an initial slight rise in elongation, as well as in proof strength and ultimate tensile strength, shown by material which had been cold worked before ageing.

Monel Buckets in Salt Driers

See abstract on p. 53.

Reactor Materials and Design

See abstract on p. 28.

Nickel Silver in Watches

'Watches: Metal Working in Miniature.'

Iron Age, 1955, vol. 176, Dec. 29, p. 27.

Popular note on the activities of Hamilton Watch

Company, Lancaster, Pa., with emphasis on the team-work investigation involved in development and perfecting of new models. Particular attention is directed to the intricacy of the watch base plate, which contains over 40 accurately sized and placed holes and 15 cuttings or recesses. From start to finish, 115 operations are involved in production of the plate, which is of 12 per cent. nickel silver, rhodium plated to enhance resistance to corrosion. The Hamilton Company makes and treats its own hairsprings from a nickel-iron-base alloy specially developed for this purpose. It is stated that 73 operations go into making these springs, some of which are as small as 0·0035 in. wide and 0·00075 in. thick.

Joining of Nickel and Nickel Alloys

MOND NICKEL CO., LTD.: 'Joining of Some Nickel Alloys.' *Publn.* 997,* 1956; 4 pp.

Reprint of leading article from *Nickel Bulletin*, 1955, vol. 28, No. 11, pp. 172–3.

NICKEL-IRON ALLOYS

Slip Patterns in Deformed Nickel and Nickel-Iron Alloy

T. NAGASHIMA and T. YAMAMOTO: 'Slip Patterns of Deformed Permalloy.'

Acta Metallurgica, 1956, vol. 4, Jan., pp. 94–7.

A recent paper in this journal pointed out an essential difference between slip patterns of the pure metals aluminium, copper and magnesium and slip patterning on alpha brass. The present note records observation of similar differences between patterns typical of slip in pure nickel and in 80 per cent. nickel Permalloy.

Magnetic Domains in Nickel-Iron Films

C. A. FOWLER and E. M. FRYER: 'Magnetic Domains in Thin Films of Nickel-Iron.'

Physical Rev., 1955, vol. 100, Oct. 15, pp. 746–7.

Employing the magneto-optic method of domain observation described in an earlier paper (*ibid.*, 1954, vol. 94, p. 52), the authors photographed the domain structure of an evaporated film of 80 per cent. nickel nickel-iron alloy 5,000 Å thick. As was anticipated, the examination revealed that each domain extended completely through the film. Sensitivity of the domains to external magnetic fields is illustrated by three typical photographs from a series indicating that the changes caused by the small field of the S pole of a small compass needle brought close to, and removed from, the film, occur by movement of the domain walls. Proximity of an unmagnetized steel needle caused a smaller, but still significant, domain shift, and when the film was left overnight domain configuration altered, due, it is believed, to small stray fields in the laboratory.

Investigation of two other films of the same material, having, respectively, thicknesses of 10,000 and 20,000 Å, showed no evidence of surface domains, but it is considered that normal domains may, nevertheless, have been present, and the matter is being further studied.

* *We shall be pleased to supply a free copy of this publication.*

CAST IRON

Specification for S.G. Iron

AMER. SOC. TESTING MATERIALS: 'Standard Specifications for Nodular Iron Castings.'

A.S.T.M. Designation A.339–55.

A.S.T.M. Standards, 1955; Part I, *Ferrous Metals*, pp. 1398–401.

This specification, issued as Tentative in 1951, has been adopted, without modification, as Standard. The mechanical-property requirements are as follows:

	Grade 80–60–03	Grade 60–45–10
Tensile strength, min. ..	80,000 p.s.i. (36 t.s.i.)	60,000 p.s.i. (27 t.s.i.)
Yield Strength, min. ..	60,000 p.s.i. (27 t.s.i.)	45,000 p.s.i. (20 t.s.i.)
Elongation in 2 in., min. ..	3·0%*	10%

* Where strength is the primary requirement the elongation requirement in grade 80-60-03 may be waived by agreement between manufacturer and purchaser. It is intended that the properties of the 60-45-10 grade shall be obtained by a graphitizing annealing treatment, but, by agreement between manufacturer and purchaser, castings are to be deemed acceptable if the properties specified are attained without heat-treatment.

Nickel-containing Materials in Marine Engineering

See abstract on p. 40.

CONSTRUCTIONAL STEELS

Nickel-Molybdenum Die Steel

J. A. SUCCOP: 'Development of a Die Block for Closed Die Forging.'

Steel Processing, 1955, vol. 41, Oct., pp. 621–35.

In this paper, presented before the American Society for Metals, lengthy consideration is given to the requirements of die-block steel and to developments sponsored by the Heppenstall Company. For many years past a chromium-molybdenum-vanadium steel, known as Hardtem, has been successfully employed, and some particulars are given of the methods used in production of drop-forge die blocks from this steel.

Modern developments in mechanical presses have,

however, necessitated the use of material of rather different type for dies. These presses have a forging action entirely different from that of drop hammers and this different forging action imposes stresses of another kind. The drop-hammer die is subjected to a heavy impact blow: the die strikes the hot steel being forged at a velocity of about 20 ft. per sec. In consequence, the hot metal remains in contact with the die surfaces for a comparatively short time. The crank press, on the other hand, delivers a positive-pressure blow which, by comparison, is also static, and the hot metal remains in contact with the surfaces of the die for a relatively long time. Constant heating and cooling under such conditions frequently results in heat checking. It is therefore obvious that steel used for press dies must have the following characteristics:—(1) resistance to thermal fatigue or heat checking, (2) resistance to hot abrasion by the forging stock and its oxide film, (3) resistance to distortion during heating and cooling involved in the forging operation, and (4) sufficient mechanical strength to prevent failure by fracture during operation.

To meet these conditions, the Heppenstall Company has developed a new type of steel, based on a low-carbon, 3 per cent. molybdenum composition, but containing nickel, which gives the required modification of structure and properties essential for press-die work. The composition limits of the steel are as follows:—carbon 0·18–0·23, silicon 0·20–0·35, manganese 0·63–0·78, nickel 3·00–3·25, molybdenum 3·25–3·50, per cent. The steel is age-hardening, and the effect of ageing on hardness at working temperatures is illustrated in this paper, showing that the steel maintains its hardness at operating temperatures even above 1200°F. (650°C.). The nickel-molybdenum composition is also characterized by marked resistance to thermal shock, such as is involved by water cooling of a die followed by rapid heating by contact with hot-forging stock.

This type of die, which has the further advantage of being readily machinable at comparatively high hardness, is now in extensive use in steel works in the U.S.A. and is covered by a U.S. patent. The steel is marketed under the name of Prestem.

Nickel-Alloy Steel Gears

'Automobile Gearbox Factory.'

Engineer, 1955, vol. 200, Dec. 30, p. 943.

Description of some of the outstanding features of the new gearbox factory recently put into commission at the works of David Brown and Sons (Huddersfield), Ltd., who have for more than forty years specialized in this type of product. The Company manufactures sixty variations of sixteen basic gearbox models, and although production is varied and adaptable, to suit special requirements, the gearboxes fall generally into two categories, those for light commercial vehicles or for heavy transport vehicles. Gear-boxes for Aston Martin and Lagonda cars are also made. Gear parts in the David Brown works are made in nickel-alloy case-hardening steels to B.S. En 36 (3 per cent. nickel-chromium) or En 39 (4½ per cent. nickel-chromium-molybdenum) steel. Equipment used in the shop in which these parts are heat-treated is described. Some details are also given of methods employed in gearbox assembly, and in inspection testing.

Nickel-containing Materials in Marine Engineering

S. F. DOREY: 'Ferrous Materials in Marine Engineering.' Twenty-Fourth Andrew Laing Lecture to the North-East Coast Institution of Engineers and Shipbuilders.

Trans. N.E. Coast Instn. Engineers and Shipbuilders, 1955, vol. 72, Pt. I, pp. 27–56.

Progress in development of ferrous materials used in this field is traced, with particular reference to that which has taken place during the twenty-five years since the death of Andrew Laing. Materials currently used for various components are reviewed, with comment on their properties, advantages, and limitations, and some views are expressed with regard to probable future trends.

The subject is dealt with in sections relating to individual components, with discussion of the types of steel and, in some cases, cast iron, used for them. On this basis the following parts are reviewed:

- shafting (for general purposes, crankshafts, rotor shafts)
- reduction gearing
- pistons
- turbine blading (in steam and gas turbines)
- stationary turbine parts
- steam plant
- propellers.

The final section of the paper covers a brief general consideration of conditions involved in service in sea water, and the degree of resistance offered by various materials to the corrosion/erosion/abrasion attack involved.

Descaling of Steels and Alloys after Exposure to High-Temperature Water

See abstract on p. 51.

Specification for Carbon and Alloy Steel Plate, Sheet and Strip

BRIT. STANDARDS INSTN.: 'Steel Plate, Sheet and Strip.' *B.S.* 1449: 1956; 36 pp. Price 6/-.

The first edition of this Standard was issued in 1948, to replace several existing schedules and to provide a more comprehensive set of specifications for steels in the form of plate, sheet and strip used in the automobile and general-engineering industries. It was parallel to the coverage of steel bars, billets and forgings provided by B.S. 970.

The present revision (which is associated with the contemporary revision of B.S. 970), brings the requirements for the automobile and general-engineering steels up to date, and also covers those of the Services, as far as they are compatible with those of industry in general.

The general clauses of the specification deal with

permissible steelmaking processes, freedom from defects, chemical analysis and testing, inspection, finish, and tolerances. Part 2 covers specific requirements for the following materials, in the forms specified:—

En 2	Ordinary quality steel plate, sheet and strip.
En 2A, En 2A/1	Deep-drawing and extra-deep-drawing steel plate, sheet and strip.
En 2B	20-ton steel plate, sheet and strip.
En 2C, En 2C/1	26-ton steel plate, sheet and strip.
En 2C/2	24-ton steel plate, sheet and strip.
En 2D, En 2D/1	28-ton steel plate, sheet and strip.
En 5	'30' carbon steel plate and sheet.
En 14A	Carbon-manganese steel plate, sheet and strip.
En 21A	3 per cent. nickel steel plate and sheet.
En 43G, 43J, 42E, 42F, 42G, 42J, 44D, 44E	Cold-rolled carbon spring steel strip and flat wire.
En 56A, 56B, 56C, 56D	Chromium rust-resisting steel sheet and strip.
En 57	Martensitic chromium-nickel rust-resisting steel sheet and strip.
En 58A, 58B, 58C, 58D, 58E, 58F, 58G, 58H, 58J	Austenitic chromium-nickel rust-, acid- and heat-resisting steel sheet and strip.
En 60, 61	Ferritic chromium rust-resisting steel sheet and strip.

Corrosion of Car Bodies: Resistance of Plain and Alloy Steels

See abstract on p. 53.

Transformations in Nickel-Alloy Steel in Relation to Weldability

B. J. BRADSTREET: 'Continuous-Cooling Transformation Diagram and Weldability of a 2¼% Ni-Cr-Mo Steel.'

Brit. Welding Jnl., 1955, vol. 3, Jan., pp. 30–2.

This report is a continuation of a series giving results of investigations made in the laboratories of The British Welding Research Association. Transformation characteristics, determined by means of continuous-cooling diagrams, have been correlated with welding properties, with particular reference to conditions which will ensure immunity from cracking in the heat-affected zone of constructional steels. (Abstracts of some earlier papers in the series will be found in *Nickel Bulletin*, 1954, vol. 27, No. 10, p. 195, and 1955, vol. 28, No. 11, p. 190.)

The present paper relates to a steel conforming to B.S. En 25: the composition of the bar used in the tests is given as carbon 0·33, silicon 0·19, manganese 0·70, sulphur 0·010, phosphorus 0·018, nickel 2·52, chromium 0·66, molybdenum 0·48, per cent. The hydrogen content is reported as 0·05 ml./100 g. A continuous-cooling diagram was constructed from the results of dilatation curves on ¼-in. long tubular specimens heated rapidly in a h.f. heating coil to a peak temperature of 1340°C., and cooled by an argon gas quench, using six different cooling cycles. Correlation of these data with the results of weld-cracking tests on metal-arc welds, made by the C.T.S.* test developed by the Association, led to the following conclusions:—

(1) For the normal type of hard-zone cracking, the critical end-of-transformation temperature, using rutile-covered austenitic (18–8 type) electrodes, was in the range 155°–175°C.

(2) Using austenitic electrodes, the critical cooling rate at 300°C. for the 2¼% Ni-Cr-Mo steel was in the range 4°–7°C./sec. This demands a preheat temperature of about 150°C. when making a ⅜-in. leg-length fillet weld (4 S.W.G. electrode, 12-in. run length) on very thick plate, but no preheat for ½-in. thick plate.

(3) Using low-hydrogen electrodes, a preheat temperature of about 200°C. was required to prevent hard-zone cracking in a ¼-in. leg-length fillet weld (8 S.W.G. electrode, 11-in. run length) on ½-in. plate.

(4) Even with slower cooling rates than those specified, small cracks occurred in some of the welds in the heat-affected zone at the toe of the fillet. The reason for this form of cracking has not yet been established.

Heat-Treatment of Welded Alloy Steels

INTERNATIONAL INST. WELDING: 'Heat-Treatment of Welded Alloy Steels.'

Brit. Welding Jnl., 1956, vol. 3, Feb., pp. 52–3.

At the 1954 meeting of the International Institute of Welding, Commission X of the Institute (concerned with Residual Stresses and Stress-Relieving) considered a lengthy document correlating reports on practices adopted in various countries for heat-treatment after welding. The length of the correlated record was too great to permit publication in extenso, and it was therefore decided to summarize the information contained in it. This summary, in tabular form, is now presented. The information covers thirteen types of steel, ranging from low- and medium-alloy† grades to the high-chromium and 18–8 chromium-nickel stainless steels.

The following comment is made by the members of the Institute who have been responsible for review of the information supplied from the member countries:

'In considering the available data the opinion has been formed that the stress-relieving treatments reported as being in use are not based on accurate experimental evidence. For purely stress-relieving purposes, there was little to show that laboratory data had been available or had been used. Where the temperature for post heat-treatment was based

**Controlled thermal severity.*
†No nickel-containing types.

on tempering of the heat-affected zone, the lack of precision in the temperatures used would seem to indicate that accurate data had not been accumulated and that, whilst no doubt considerable latitude might quite rightly be permissible in practice, it would be preferable if general agreement could be reached upon fundamental and reliable data.

'Lastly, though not apparent from the final summary, it appeared that the precise technical benefits claimed from stress-relieving had not been substantiated by experimental evidence, and were, in fact, primarily matters of opinion.

'The conclusion of the Sub-Commission is therefore that, whilst in the majority of cases satisfactory heat-treatments are specified, and whilst the choice has been based on sound metallurgical principles, precision is lacking because the fundamental data are not generally available.

'In recognition of this fact the Commission has initiated extensive experimental investigations with a view to obtaining such fundamental data.'

Nickel-Alloy Hard Facing on Compressor Rods

See abstract on p. 54.

HEAT- AND CORROSION-RESISTING MATERIALS

Precision Forging of High-Nickel Alloy Turbine Blades

E. B. WELLS: 'Production of Gas-Turbine Blades by Precision Forging.'

Machinery (*Lond.*), 1955, vol. 87, Dec. 2, pp. 1292–7.

At the works of Utica Drop Forge and Tool Corporation, U.S.A., precision forging is being extensively used for production of gas-turbine blades varying from 2 to 12 in. in length. The accuracy with which dimensional requirements can be met in the finish-forged blade lessens waste of material and reduces machining to a minimum. For compressor blades, aluminium alloys or stainless steels of the straight-chromium series are used: for gas-turbine rotor blading a wide range of high-temperature alloys is handled, including those of the Nimonic series, cobalt-base alloys, 16-25-6 chromium-nickel-molybdenum steel, and other materials which are in large-scale use for this application. An unusual feature of the facilities at the Utica plant is that vacuum furnaces are used for melting a substantial proportion of the heat-resisting alloys, and it is found, for example, that stress-rupture of Waspaloy blading made from vacuum-melted heats is markedly superior to that obtainable from blading produced from electric-arc-melted material made by conventional methods. Two 1,000-lb. vacuum units in this plant are believed to be the only ones now in operation which provide for centrifugal casting.

Heating, forging, finishing and inspection procedure are outlined in this article, with illustrations of some of the equipment used.

Nickel-containing Alloys in Aircraft Gas Turbines

'Bristol Olympus. The Western World's Most Powerful Production Turbojet.'

Flight, 1955, vol. 68, Dec. 9, pp. 869–76.

Record of the development of this engine, from 1946 onwards: a detailed drawing shows the main constructional features. The Bristol Olympus is the first aircraft gas turbine to have two independent axial compressors in series, and in connexion with the selection of this design a summary is made of the advantages to be gained by the use of compounded axial compressors. The individual types of Olympus which have emerged from the original form are reviewed, and some comments are made on American engines of comparable types.

'The Rolls-Royce Avon Family.'

Aeroplane, 1955, vol. 89, Dec. 16, pp. 944–57.

A historical survey of the evolution of the Avon-type engines, from the basic conception of design in the immediate post-war period, through to the development of the R.A. 21, the most up-to-date version of the Avon for which information is available.

'Armstrong Siddeley Sapphire. An Outstandingly Powerful and Efficient Single-Shaft Turbojet.'

Flight, 1956, vol. 69, Jan 6, pp. 17–22.

Historical review, from the earliest stages of design by Metropolitan Vickers Electrical Company, Limited, through large-scale development by Armstrong Siddeley, Limited, to adoption in the U.S.A. as well as in Britain. Emphasis is laid on the simplicity of design of the Sapphire engine.

In all three articles reference is made to the extensive use of high-nickel alloys in the units described, e.g., for rotor blading, flame tubes, and other components.

Creep-Rupture Properties of 18-8 Chromium-Nickel Steels

F. C. MONKMAN, P. E. PRICE and N. J. GRANT: 'The Effect of Composition and Structure on the Creep-Rupture Properties of 18-8 Stainless Steels.'

Trans. Amer. Soc. Metals, 1955, vol. 48, *Preprint* 6; 34 pp.

The relatively high cost and limited availability of many elements currently used in high-temperature alloys is resulting in much interest in the possibility of improving the properties of steels of lower alloy content. This paper reports a basic investigation of the '18-8' chromium-nickel steel series, as offering promise in this connexion: the aim of the work was to develop a more accurate knowledge of the influence of variations in composition (within the specification limits usual for this type of steel) on stability, high-temperature strength, and creep-resistance.

The tests were made on a series of simple chromium-nickel stainless steels containing carbon and nitrogen, but without addition of other alloying and stabilizing elements usually found in the commercial steels of this type. Since previous work had indicated

a strong dependence of rupture strength on the carbon+nitrogen content of unstabilized 18–8 steels, the sum of these two elements was kept constant when the nickel or chromium was varied. Twenty-seven steels were used, varying, within the broad range, chromium 19·78–10·71, nickel 6·09–18·38, per cent.

Stress-rupture tests were made at 1000°, 1200° and 1300°F. (540°, 650° and 705°C.) and at stresses which resulted in rupture lives from about 30 seconds to 500 hours. Metallographic samples were prepared from selected stress-rupture specimens for observation of changes in structure occurring during testing. A high-temperature X-ray camera was used to determine structure at test temperatures, and formation and reversion of martensite was studied by means of resistivity measurements. In addition, the amount of magnetic phase present in the alloys was estimated by a Magnegage.

The results of the respective series of tests are shown in graphs giving log stress *vs.* log rupture life at the three temperatures of test, supplemented by photomicrographs of stress-rupture specimens.

The observations made lead to the following conclusions:—

Increasing amounts of *carbon and nitrogen* in solid solution strengthen stainless steels at elevated temperatures, both by alloying the austenitic matrix and by suppressing formation of sigma and ferrite, but both carbon and nitrogen ultimately tend to precipitate out of the solid solution, as carbides and nitrides, and the strengthening effect is therefore time- and temperature-dependent. At 1200° and 1300°F. (650° and 705°C.) precipitation was relatively rapid: at 1000°F. (540°C.) the reaction was much slower, and at that temperature steels of increased carbon+nitrogen content showed significant improvement in strength up to 500 hours or longer. The effect of these two elements in stabilizing the austenitic matrix was beneficial in counteracting the influence of ferrite growth at the grain boundaries. Where ferrite occurred, its initial formation enhanced high-temperature strength, but its subsequent growth and agglomeration had a weakening effect. Instability in the stress-rupture life curves liable to be caused by precipitation of sigma was suppressed by increasing the carbon+nitrogen content of the steels.

Chromium also affects high-temperature strength, both as an alloying element in the austenitic matrix, and by influencing structural changes. At the test temperatures used, its strengthening effect, although important, was less potent than that of carbon and nitrogen: there was, however, the important advantage that the beneficial influence of chromium did not diminish to any great extent with time at temperature. This element was detrimental when it promoted sigma formation: initial precipitation was associated with strengthening, but subsequent growth of sigma at the grain boundaries had a severely weakening effect.

The finding of the authors is that *nickel* has no important strengthening influence on high-temperature rupture properties.

The martensitic ferrite which formed in these steels by cooling below the M_s temperature had no significant effect on rupture and creep properties at 1000°F. (540°C.), but decomposition of martensite occurring above 1100°F. (595°C.) caused a fall in strength and ductility at 1200° and 1300°F. (650° and 705°C.).

Ductility decreased with increasing life-to-rupture. This property was essentially independent of both chromium and nickel contents in the range studied, but varied inversely to the carbon+nitrogen content.

Influence of Environment on Behaviour of Metallic Materials at High Temperatures

AMER. SOC. TESTING MATERIALS: 'Symposium on Basic Effects of Environment on the Strength, Scaling and Embrittlement of Metals at High Temperatures.'

A.S.T.M. Special Technical Publn. 171, 1955; 114 pp. Published by the *Society, Philadelphia, Pa., U.S.A.;* Price $2.75.

The publication contains the reports and discussions presented at a symposium held in Cincinnati in February 1955.

E. A. DAVIS: 'Introduction', p. 1.

Most of the creep and creep-rupture data on which design can be based have been obtained from tests carried out in normal atmospheres. This symposium was organized with the hope of summarizing the present state of knowledge on modification of properties which might result from the varied types of contaminated environment in which parts may be called upon to work at elevated temperatures.

J. J. GILMAN: 'The Rôle of Thin Surface Films in the Deformation of Metal Monocrystals.' pp. 3–11; disc., pp. 11–13.

Data available in the literature show that the strengthening effects of surface films on metal monocrystals (the 'Roscoe effect') cannot be explained in terms of the strength of the film itself. It is believed that interaction between the film and the crystal surface must be involved. It is concluded that the Roscoe effect is caused by the inhibiting effect that a film has on the exit of slip from a crystal. The effect that a film might have on the generation of slip on the surface is believed not to be an important factor.

J. F. RADAVICH: 'Structure of Oxides Formed on High-Temperature Alloys at 1500°F. (815°C.).' pp. 14–31; disc., pp. 32–4.

The author reports studies of the nature of the oxide films formed, with and without the presence of stress, at 1500°F. (815°C.), on alloys of the following types:—

19-9 DL—18–8 type chromium-nickel steel containing 1 per cent. molybdenum, and about 2·5 per cent. tungsten.

Type 347 Steel—Niobium-stabilized steel containing chromium 18, nickel 11, per cent.

Type 17–14 Chromium-nickel steel containing copper and molybdenum.

Type 310 Steel—Chromium 24, nickel 21, per cent.

N - 155 Multimet — Complex nickel - chromium - cobalt-iron alloy containing molybdenum, niobium and tungsten.

Hastelloy C—Nickel-molybdenum-chromium alloy containing about 4·5 per cent. each of iron and tungsten.

Type 316 Steel—Niobium-stabilized steel containing chromium 18, nickel 14, molybdenum 2, per cent.

X-ray analysis of the oxide scales formed during stress-rupture tests at 1500°F. (815°C.) showed a strong rhombohedral αFe_2O_3 phase on all the austenitic alloys except the 24-21 per cent. chromium-nickel steel: this difference may, it is believed, be due to the high chromium content of this steel. A small amount of spinel phase was found in the scale on all the austenitic materials. Relatively few differences were found between the oxidation characteristics of the stressed and the unstressed, specimens.

Examination of the oxide film initially formed, after five minutes at 1500°F. (815°C.) indicates that amorphous silica may be associated with the thin oxide films which are next to the metal. It is suggested that if this substance is present in the thin films, it may be a very influential factor with regard to the behaviour of the metal at elevated temperatures. It is noted that in a recent investigation on the oxidation of austenitic and ferritic stainless steels at (1800°F.: 980°C.), i.e., a temperature higher than that used in the present study it was observed that a continuous amorphous-silica layer was present at the temperature of oxidation. Since amorphous silica has been used in the polystyrene-silica replica technique (due to its high mobility on the polystyrene surface), a similarly high mobility may be associated with an oxide surface. Although no positive conclusion can be drawn from the present work, it is considered possible that the action of amorphous silica as a healing agent may be greater than that of the oxide scale or of films formed at the temperature of oxidation.

Macroscopic examination of the specimens used in this investigation indicated a correlation between the number of cracks in the metal and the number of cracks in the oxide scale. There was no apparent correlation between the number of cracks in the metal and scale and the stress-rupture characteristics of the materials, but it is considered that this absence of relation may be due to variation of stresses used in testing, since some correlation was observed between the cracks in the oxide and metal and the stress-rupture life of materials in a similar stress range. More cracks were found in the base metal than in the oxide scale.

E. A. GULBRANSEN and K. F. ANDREW: 'Effect of Strain on the Oxidation of Nickel-Chromium Heater Alloys', pp. 35-46; disc., 47-8.

Methods used for evaluation of the oxidation-resistance of metals and alloys can be classified into two groups.

(1) Those which utilize measurement of the amount of reaction at constant temperature, e.g., direct weighing technique, change of volume or pressure in the reaction vessel, or measurements of some physical property of the oxide or of the sample.

(2) Those involving oxidation under conditions in which the sample is alternately heated and cooled. As in the first method, the amount of reaction may be determined in several ways.

In the A.S.T.M. life test for heater alloys (which falls into group 2), a wire specimen is heated for two minutes to 1175°C., and then cooled for two minutes before re-heating. Heating is done electrically, at constant voltage. Time, in hours, for a 10 per cent. change in electrical resistance gives a measure of oxidation, and is termed the 'useful life' of the resistance element.

Previous work on test methods is reviewed by the present authors, and reference is made to the recent development, by them, of a test* in which the alloy can be strained at an intermediate stage during oxidation. Comparison is made of the rate of oxidation before and after straining.

Preliminary results showed that in this strain-oxidation test heater alloys having high A.S.T.M. useful lives showed no deleterious effects due to strain: rate of oxidation actually decreased after straining. Heater alloys having low A.S.T.M. useful lives, have however, shown an appreciable increase in rate of oxidation as a result of strain. In the present paper the strain-oxidation test is applied to a series of 80-20 nickel-chromium heater alloys, and the results are correlated with A.S.T.M. useful lives and with results of oxidation tests at constant temperature.

The programme was drawn up with four main objectives:—

(1) To determine the effect of a 2 per cent. strain on the behaviour of a series of nickel-chromium alloys containing variable silicon and manganese.

(2) To ascertain the effect of initial thickness of oxide layer on the strain-oxidation behaviour of low- and high-silicon nickel-chromium alloys.

(3) To determine the effect of amount of strain on the strain-oxidation behaviour of the variable-silicon alloys.

(4) To demonstrate the strain-oxidation behaviour of high- and low-silicon nickel-chromium alloys at 950°C.

Full details of composition of the alloys tested are given.

The results showed that two factors are of primary importance in oxidation tests in which the metal-oxide system is subjected to stress: the first is the constant-temperature rate of oxidation, the second the behaviour of the alloy in relation to stress while undergoing reaction.

Two types of damage in the oxide film were found to result from strain: the first is shown by a rapid

**Details of this test are to be published shortly, in a paper on 'The Rôle of Minor Elements on the Oxidation of Nickel-Chromium Heater Alloys.'*

rate of oxidation on first reoxidizing the metal, which is rapidly repaired during further oxidizing, whereas the second is a long-term effect, which may result either in permanent damage or in improvement in oxidation behaviour.

Using a 2 per cent. strain, it was found that the low-silicon nickel-chromium alloys showed more damage from strain than those of higher silicon content, a result which is in accordance with the A.S.T.M. useful lives of the respective materials. The amount of strain was proved to be a significant variable, as was the oxide-film thickness at which the strain was imposed on the specimens. For films of 2,600 Å none of the alloys were damaged in the long-term sense by a 1 per cent. strain, and for thin oxide films of about 1,700 Å none were damaged by a 2 per cent. strain. A strain of 4 per cent. for a film thickness of 2,600 Å caused no damage with the high-silicon alloys. It is, however, of interest to note that the high-temperature rate of oxidation of the higher-silicon materials was much greater than for those of lower silicon content, suggesting that silicon increases the rate of oxidation of nickel-chromium alloys at a constant temperature. The mechanism of this effect is discussed.

R. E. KEITH, C. A. SIEBERT and M. J. SINNOTT: 'An Investigation of Intergranular Oxidation in Type 310 Stainless Steel', pp. 49–60; disc. pp. 60–4.

Report of investigation undertaken to establish the effects of time and temperature on intergranular oxide penetration, and to determine whether appreciable differences in composition exist between the intergranular oxides and the external scales.

Since one of the main objectives of the research was to determine the effects of minor variation in composition, specimens from a number of different heats were studied, including one of Type 309 steel (carbon 0·20 max., chromium 22–24, nickel 12–15, per cent.) stabilized with niobium, six heats of Type 310 steel (carbon 0·25 max., chromium 24–26, nickel 19–22, per cent.), and two modified Type 310 compositions.* The six normal 310 steels were selected to show as wide variations in composition as was possible within the A.I.S.I. specification limits. Oxidation treatment was in dry moving air, at temperatures between 1600° and 2000°F. (870°C. and 1095°C.) for 10, 30 and 100 hours. All the test specimens were in the form of cold-finished sheet, 0·04–0·06 in. thick.

Microscopic examination of the steels, and investigation of the scales by X-ray diffraction and of the intergranular oxides by reflection electron diffraction, led to the following conclusions:—

(1) Depth of intergranular oxide penetration was not appreciably affected by minor variations in the composition of the steels.

(2) Depth of oxide penetration increased with time and temperature of oxidation: temperature was the more significant factor.

(3) The external scales consisted of a rhombohedral phase $Fe_xCr_{2-x}O_3$ and up to two different spinel phases, believed to be $Fe(Fe_xCr_{2-x})O_4$ and $MnCr_2O_4$.

(4) So far as could be determined, the compositions of the intergranular oxides were identical with those of the corresponding external scales.

The authors propose a mechanism of intergranular oxidation in which the movement of oxygen is defined as being a case of conventional intergranular diffusion. The formation of the intergranular oxide is not controlled by grain-boundary diffusion: it probably depends on the presence of suitable metal ions and nucleation sites, as well as on the presence of oxygen.

One containing lower nickel, the other lower in both chromium and nickel.

M. GLEISER, W. L. LARSEN, R. SPEISER and J. W. SPRETNAK: 'The Properties of Oxidation-Resistant Scales Formed on Molybdenum-base Alloys at Elevated Temperatures', pp. 65–86; disc., pp. 87–8.

It is now generally recognized that one of the chief deterrents to the use of molybdenum and its alloys as high-temperature materials is the susceptibility of this metal to oxidation at high temperatures. The present authors direct attention to comprehensive surveys of the problem which have appeared in the literature.

Although protection by cladding with certain oxidation-resisting materials has been found to some extent efficient,* it is obvious that 'barriers' resulting from alloying molybdenum with other metals would have one significant advantage over clad materials, in that the alloy surfaces, or compounds formed by them, would be capable of being self-regenerative in the event of cracking or rupture caused by thermal or mechanical stresses, and that if the alloy had a uniform concentration of the added component there would be less likelihood of destruction of the coating by inward diffusion. Based on such argument, an extensive programme was therefore drawn up to cover study of oxides or molybdates such as might be produced from alloys containing molybdenum. Since the critical factor would be the properties of the compound formed on the alloy surface, the initial investigation comprised tests on the properties of a wide range of molybdates, with a view to subsequent trials on alloys which would produce such compounds. The properties by which the molybdates were evaluated were solubility, hygroscopic or deliquescent nature, melting point, weight stability during heating, density, and crystal structure.

Examination of 22 molybdates, by such methods, showed that several of these compounds merited consideration, but subsequent oxidation tests on alloys of molybdenum which would furnish such compounds narrowed the range of study to alloys of molybdenum with cobalt or nickel, and the major work reported in this paper was done on alloys of these two groups.

A very extensive series of experimental results is recorded, and in view of the novelty of the approach,

*e.g., see abstract on p. 47.

and the practical importance of the results, the conclusions reached by the authors are reported in extenso:—

Cobalt molybdate and nickel molybdate occur in three allotropic forms between room temperature and 900°C. These allotropes occur in the same order in the two types of molybdate and are crystallographically isomorphous. Transformation of the molybdates from the γ to the β form is associated with physical disruption, designated 'spalling'. This effect may be suppressed by lowering the transformation temperature, which can be accomplished by adding certain elements contained in a γ-phase solid solution.

Molybdenum-nickel alloys containing 30 wt. per cent. of nickel were found to be completely protected from oxidation at 940°C., after formation of a thin protective scale of nickel molybdate ($NiMoO_4$). The scale of these alloys is, however, partially destroyed by spalling on cooling, unless the γ phase is stabilized by addition of a third component in solid solution. In the cobalt series, alloys containing more than 20 wt. per cent. of cobalt proved to be resistant to oxidation at the same temperature, after formation of a thin scale of cobalt molybdate ($CoMoO_4$). In this case also, however, it is necessary to prevent spalling by addition of a third component. Further tests demonstrated that manganese dioxide or magnesium molybdate, added to the powdered cobalt or nickel molybdates, can depress the γ-β transformation below room temperature, and similar γ-phase stabilization occurs in the scale formed on the surface of molybdenum-cobalt-manganese and molybdenum-nickel-manganese alloys oxidized at 940°C.

Temporary stabilization of cobalt-molybdate powder was achieved by addition of silica, and a molybdenum-cobalt-silicon alloy also formed a protective stabilized γ-phase scale for about 200 hours at 940°C., but after that time protection failed and the scale became unstabilized. Cobalt molybdate and nickel molybdate reacted at temperatures above 1000°C. with equimolar amounts of alumina, chromic oxide or ferric oxide, liberating molybdic oxide, and resulting in the formation of spinel-type oxides $CoAl_2O_4$ or $NiAl_2O_4$, $CoCr_2O_4$ or $NiCr_2O_4$, and $CoFe_2O_4$ or $NiFe_2O_4$, respectively. A similar reaction took place on the surface of a molybdenum-nickel-iron alloy at 1110°C., but was not observed at 950°C. on molybdenum-nickel-chromium or molybdenum-cobalt-aluminium alloys. In the latter two cases unreacted chromic oxide and alumina, respectively, were found in the scale.

It is concluded that the approach to the problem of protection studied by the authors has been fully justified by the results obtained with the molybdates of nickel and cobalt, and that further research should be carried out to amplify the data so secured.

Among the aspects still requiring investigation the following are mentioned:—

(*a*) The crystal structure of molybdates.

(*b*) The stabilization of β or γ forms of cobalt and nickel molybdates.

(*c*) Binary phase diagrams of molybdates with oxides of possible alloying elements of molybdenum.

(*d*) Elements other than nickel and cobalt which might form stable molybdates as a result of alloying with molybdenum.

(*e*) Oxidation protection by 'sacrificial oxidation' of molybdenum.

(*f*) Use of oxidation-resistant molybdenum alloy base metal or of a thin layer of an oxidation-resistant alloy over the molybdenum base metal, in conjunction with ceramic or metallic claddings.

It is noted that some of these subjects are already under investigation by the present authors.

In discussion, the question of the high-temperature mechanical properties of the modified materials was raised. This aspect will be separately studied, but attention was directed to the fact that an alloy containing nickel 17·3, aluminium 2·7, per cent., molybdenum balance has already been the subject of patent claims, as combining forgeability with oxidation-resistance (*U.S. Patent* 2,698,786).

J. F. RADAVICH: 'Oxidation at Elevated Temperatures', pp. 89–113; disc., pp. 113–14.

Investigations on ferritic stainless steels (5–26 per cent. chromium) have shown that initial oxidation takes place by formation of a thin oxide substrate on which nodules of oxide nucleate and grow, and preliminary work on austenitic alloys oxidized at 815°C. has indicated that the mechanism of growth is similar on most of the commercial austenitic alloys, but that it differs in the case of the high-temperature alloys N-155 and Hastelloy C.*

The present paper reports an investigation of the initial oxidation characteristics of seven high-temperature materials of the same types as those used in the experiments reported by the author in his earlier paper to this Symposium: see p. 43.

Most of the oxidation tests were made at 600°C., but some data for 815°C. tests are also included. The work is reported in two sections:—

(1) Study of initial oxide growth by means of electron microscopic examination of surface replicas and stripped oxide films. (This section includes electron-microscopic studies of the oxide growth occurring on one region after repeated oxidations of the N-155 Alloy and a typical iron-chromium-nickel composition.) The report confirms that the oxide on iron-chromium-nickel alloys grows by development of a thin film and nucleation and growth of oxide nodules, whereas on the more complex materials, such as N-155 or Hastelloy C, the oxide growth takes place as a uniform thin film.

(2) Analysis of the structure and identification of the oxide films formed on the seven alloys examined. A tabular summary is presented of the oxides formed on the respective materials after 5, 30 and 60 minutes, and 10 and 24 hours at 600°C., and after 5, 15 and 45 minutes at 815°C. The

*For type see p. 44.

observations made by the four methods of examination† used are individually recorded, and the data obtained by the respective procedures are critically correlated.

Clad Molybdenum Sheet: Production and Properties

M. H. LACHANCE and R. I. JAFFEE: 'Fabrication and Evaluation of Thin Clad Sheets of Molybdenum.' *Trans. Amer. Soc. Metals*, 1955, vol. 48, *Preprint* 43; 34 pp.

The purpose of the work described was to devise methods for production of thin clad molybdenum sheet capable of giving limited service life in air at elevated temperatures. The criterion set up for 'satisfactory' behaviour was adequate protection, by the cladding, during service at 1800°, 2000° or 2200°F. (980°, 1095° and 1205°C.) for (*a*) 100 hours at constant temperature, and (*b*) 40 one-hour cycles from the test temperature to room temperature.

Cladding Procedure

Initial efforts were concentrated on (*a*) development of a cladding technique which would ensure good metallurgical bonding, and (*b*) perfecting a method of edge sealing the sheared composite sheet. It was found that good bonding could be effected by sealing the cladding assembly under vacuum prior to rolling. The tendency of oxidation-resistant alloys containing chromium to form stable surface oxides when heated in air proved detrimental to bond adherence: even in an edge-welded assembly sufficient air was occluded to allow such films to form. Two methods were evolved to obviate this difficulty: (1) the contact surfaces of the cladding materials were coated, before application to the molybdenum sheet, with a thin electrodeposited layer of nickel, *or* (2) thin interlay sheets of nickel could be used, provided that occluded air was evacuated. The assemblies were in all cases heated in hydrogen for rolling: 2190°F. (1200°C.) was found to be the optimum temperature for this operation. After finish-rolling the composite sheet was hydrogen annealed.

In the production of clad sheets it is usual to finish-roll to the desired thickness, and shear to size. For clad molybdenum, protection of sheared edges is essential, and for the sealing of double-clad sheets the following procedure proved satisfactory:—undercutting the molybdenum core section by anodic immersion in caustic solution, inserting a filler wire in the groove so formed, and fusing the edges with an oxyacetylene torch.

Ductility Tests

Ductility of the composite sheet (14 mils molybdenum with 3 mils of nickel-alloy cladding on each side) was adequate to permit bending through a 105° arc over a $\frac{1}{64}$-in. radius without failure of the composite or the bond.

†(*1*) *Reflection electron diffraction*
(*2*) *Transmission electron diffraction*
(*3*) *X-ray diffraction*
(*4*) *X-ray spectrographic analysis in chromium: iron ratios.*

Oxidation Tests

A total of some thirty metals and alloys (some in both wrought and cast forms) were exposed (*a*) in still air at 1800°F. (980°C.) and (*b*) at the same temperature in a still-air atmosphere containing molybdenum trioxide. Based on the results of these screening tests, materials were selected for test in the form of claddings on molybdenum sheet: these comprised the following types:—

* A Nickel (nickel 99·5 per cent.).
* Z Nickel (aluminium 4·5, titanium 1, per cent., balance nickel).
* Inconel (chromium 15, iron 5, per cent., balance nickel).
* Inconel X (chromium 15, iron 7, titanium 2·5, aluminium 1, niobium 1, per cent., balance nickel).
* Nickel-Chromium Alloy (80–20 type).

Stainless Steel (containing chromium 15, nickel 20 per cent.).

Chromium Steel (chromium 25 per cent.).

* Nickel-Tantalum alloy (tantalum 5·2 per cent.)
* Nickel-Zirconium-Aluminium Alloy (zirconium 5, aluminium 2·5, per cent.).
* Nickel-Titanium-Silicon Alloy (titanium 2·3, silicon 2·7, per cent.).
* Nickel – Titanium – Silicon – Aluminium Alloy (titanium 1·9, silicon 2·1, aluminium 1·9, per cent.).

The materials marked with an asterisk were included in both constant-temperature and cyclic tests.

Results in the screening tests showed that iron, iron-base alloys, cobalt and cobalt-base alloys were 'catastrophically oxidized' when heated at 1800°F. (980°C.) in an atmosphere of air+molybdenum trioxide. In some instances the deterioration was even more rapid when the samples were heated in physical contact with molybdenum metal. Chromium, nickel, and nickel-base alloys showed relatively high resistance to attack by the air/molybdenum trioxide mixtures.

In the tests of composite plate, three-mil. claddings of Inconel, Inconel X, and 80–20 nickel-chromium alloy on molybdenum provided good protection in 100-hour tests in still air at 2000°F. (1095°C.), and claddings of Inconel and 80–20 nickel-chromium (of similar thickness) were also protective on samples thermally cycled from that temperature.

In still-air conditions at constant temperature specimens on which the cladding had been penetrated (intentionally or by localized oxidation), leaving the molybdenum partially unprotected, were well protected by unalloyed nickel and nickel alloys containing more than 92 per cent. nickel. The ability of such claddings to protect in these conditions is attributed to the formation of a surface compound which closely resembles the high-temperature form of nickel molybdate. (In connexion with protection by nickel molybdate see paper by Gleiser *et. al.*, abstracted on p. 45.)

Sprayed Metal Coatings for Protection of Molybdenum

A. J. HERZIG and J. R. BLANCHARD: 'Protecting Molybdenum from Oxidation.'

Metal Progress, 1955, vol. 68, Oct., pp. 109–14.

This article opens with a general note on the problem of 'catastrophic oxidation' of molybdenum and the various methods which are being proposed for protection. Tests used for evaluation of protective coatings are then briefly reviewed (oxidation, thermal-shock, ballistic-impact, ductility, and erosion tests): the essential features of the respective methods are noted.

An account is given of production of sprayed coatings on molybdenum, and of tests of their efficiency, as gauged by the tests referred to above. For application of the sprayed coatings the surface of the molybdenum is roughened, a coating 0·012–0·015 in. thick is deposited by a mechanically operated spray gun, the spray-coated surface is painted with a slurry of fine alumina powder and methyl alcohol, and the coated material is heated (in purified and dried hydrogen) for two hours at 2000°F. (1093°C.) to consolidate the coating and bond it to the base. Surfaces so produced are sufficiently smooth for most applications, but, if necessary, grinding can be carried out with silicon-carbide wheels.

The compositions of the sprayed coatings used are shown in the table below.

The summary of results indicates that sprayed coatings hold promise for certain limited applications. They provide a considerable degree of protection against oxidation, and are less brittle than ceramic coatings, although less ductile than plated or clad coatings. It is concluded that spraying may be suitable for use on some large and complicated shapes which are difficult to protect by other means.

Oxidation of Stainless Steels

H. J. YEARIAN: 'Investigation of the Oxidation of Chromium and Nickel-Chromium Steels.'

Purdue Univ. Report, 1954; 98 pp.

Report of X-ray diffraction study of the oxide scales formed (*a*) on commercial steels containing 5–20 per cent. chromium, exposed for periods up to 100 hours, in air or in oxygen at 700°–1160°C., and (*b*) on alloys and steels containing chromium 0–26 per cent. with nickel 8–80 per cent., when exposed for 100 hours in air at temperatures of 870°–1205°C.

The original of this report is available on micro cards only: a summary of the observations made is given in *Metallurgical Abstracts*, 1955, vol. 23, Dec., p. 318.

Accelerated Test for Determination of Susceptibility to Attack by Vanadium and Sodium Compounds at High Temperatures

W. E. YOUNG, A. E. HERSHEY and C. E. HUSSEY: 'The Evaluation of Corrosion Resistance for Gas-Turbine-Blade Materials.'

Trans. Amer. Soc. Mechanical Engineers, 1955, vol. 77, Oct., pp. 985–94.

The authors show that change in surface condition, measured by a modified type of Brush surface analyzer, and expressed in terms of surface roughening, can be used as a means of evaluating the probable service behaviour of materials in contact with the combustion products of residual fuel oils. The accelerated test, made in apparatus which is described and illustrated, provides for exposure in corrosive conditions which parallel given conditions in gas-turbine operation. The results obtained with the surface-roughening test correlate well with operating experience in a gas-turbine locomotive and with

Composition of Sprayed Metal Coatings for Molybdenum

Coating Alloy	Si %	Cr %	Al %	Ni %	Mo %	B %	Fe %	Other Elements %
			Mixed with Aluminium for Spraying					
Cr-Si Alloy ..	40·55	55·96	0·44	—	—	—	1·40	—
Cr–Mo–Si Alloy ..	32·97	31·30	—	—	31·29	—	0·71	—
Mo–Ni–Si Alloy ..	32·17	—	1·53	30·57	37·0	—	0·22	—
Fe–Si Alloy ..	50·78	—	—	—	—	—	Bal.	—
Ni–Si Alloy ..	30	—	—	60	—	—	7	Ca 3
			Sprayed without Aluminium					
Colmonoy No. 5 ..	4·01	12·07	—	79·16	—	2·36	4·13	C 0·46
Colmonoy No. 6 ..	4·33	15·29	—	72·19	—	2·77	4·41	C 1·02
Coast Metals modified 50 Alloy ..	3·5	—	—	93·25	—	2·25	—	C 1·00

long-term laboratory tests made with combustion rigs working at atmospheric pressure.

The data obtained from all sources indicate that vanadium, in the form of V_2O_5, and sodium, probably in the form of Na_2SO_4, are the most prevalent causes of corrosion arising from the use of residual fuels. With the former compound catastrophic corrosion occurs between 1200° and 1300°F. (650° and 705°C.), and with the latter between 1600° and 1700°F. (870° and 925°C.). (Apparatus devised for ash-fusion-temperature determinations is described.)

Tests made on typical high-temperature alloys (Inconel 'X' nickel-chromium-base alloy, 25-20 chromium-nickel stainless steel, and two types of Stellite cobalt-base alloy) show that Inconel 'X' gives the best resistance to attack by vanadium pentoxide, but that it is very susceptible to corrosion by Na_2SO_4. Addition of suitable proportions of magnesium, to the fuel oil or the ash, reduced corrosion by both the vanadium and the sodium compounds. It is noted that careful selection must be made of inhibiting additives, since many which may show promise in laboratory or short-time tests may in long-term service actually increase corrosion, due to formation of excessive deposits on the turbine blades.

Oxidation of Nickel-Chromium Alloy Wires in Thermocouples

N. F. SPOONER and J. M. THOMAS: 'Longer Life for Chromel-Alumel Thermocouples.'

Metal Progress, 1955, vol. 68, Nov., pp. 81-5.

The work described in this article forms a logical sequence to that reported by the authors in 1953 (*Jnl. of Metals*, vol. 5, June, p. 844: see *Nickel Bulletin*, 1953, vol. 26, No. 8-9, p. 148).

Following the observation that preferential intergranular oxidation of chromium in 90-10 nickel-chromium alloys could occur at low levels of oxygen pressure, in tubes having extreme length-to-diameter ratios, it has been found that this effect is responsible for errors arising during service of Chromel-Alumel thermocouples operating in long narrow protection tubes.

Experiments with protected thermocouples working under varying conditions have led to the following theory to account for the change in e.m.f. of the couples.

'With an inadequate air supply (as in a tube with the cold end partially or totally closed, or in a long narrow tube, especially if insulators fill most of it), oxygen is depleted initially by normal oxidation of the thermocouple wire and of the inner surface of the protection tube.

'When the oxygen reaches a critical low partial pressure, intergranular oxidation of the thermocouple wire begins. If the tube is completely sealed this action may be supported by oxygen content in the oxidized surface of the tube, and by an oxide finish on the wire.

'According to this theory, a metal tube should produce a worse effect than a refractory tube, and an unalloyed iron or steel tube would be worse than a stainless steel tube, due to the two conditions necessary—initial oxidation and subsequent release of that oxygen to the chromium of the nickel-chromium alloy. These ideas have been verified by observation of useful thermocouple life in laboratory tests. From these conclusions it follows that if all oxygen is excluded from the tube, thermocouple life should be almost indefinite, and it is also logical that if a material such as titanium, which has a greater affinity for oxygen than has chromium, were placed inside the protection tube, preferential oxidation of chromium would be further reduced or eliminated.'

Results shown in the paper demonstrate the beneficial effect of scavenging or exclusion of oxygen by titanium used as a getter. It is suggested that it may be possible to obviate the use of a getter by means of thorough cleaning, evacuating or sealing, but it is possible that the oxygen derived from the refractory insulator alone may necessitate the use of a getter, in addition to other precautions.

Electric-Furnace Production of Stainless Steel

Among the papers presented at the A.I.M.E. Electric-Furnace Steel Conference held in Pittsburgh in December 1955 were four dealing with melting practice in the production of stainless steel:—

R. B. SHAW: 'Survey of Stainless Steel Melting Points Up Rapid Progress.' *Jnl. of Metals*, 1955, vol. 7, Dec., pp. 1309-15.

The author draws attention to the spectacular increase in stainless-steel ingot production which has taken place since 1940, commenting on some of the factors which have contributed to such progress, especially oxygen-blowing. The advantages of this procedure are considered at some length, with notes on calculation of the oxygen requirement, and other considerations which arise in large-scale production. Reactions during the reduction period, the calculation of silicon required, and the control of conditions during the finishing period are also discussed. The possibility of consistent and satisfactory production of extra-low-carbon stainless steels, by the methods now in use, is emphasized. Other aspects considered include procedure to be adopted for prevention of hydrogen absorption, and control of oxide inclusions.

D. J. CARNEY: 'Factors in Slag Reduction Stage Detailed for Stainless Melting Practice', *ibid.*, pp. 1321-3.

With the advent of the use of oxygen for decarbonization, the increased use of stainless-steel scrap, and the demand for lower carbon contents, the slag reduction stage (which is used primarily to recover chromium, manganese and iron which enter the bath during removal of the carbon) has become an increasingly important phase of melting. In this paper the author discusses the chemical, physical and operational factors involved, and makes recommendations with regard to aspects which, in association with economic considerations, must be taken into account in carrying out the slag reduction stage.

L. F. WEITZENKORN: 'Reducing Period in Melting of Stainless Steel Described', *ibid.*, pp. 1323–4.

A step-by-step account is given of practice used at the Baltimore works of Armco Steel Corporation. Details are given of materials used, timing of operations, control tests, etc.

C. YUTMEYER: 'Deoxidizers and Tapping Temperature in Stainless Steel Production', *ibid.*, pp. 1325–6.

It is noted that, in addition to the ferro-silicon and ferro-titanium used as deoxidizing media, it has become necessary, in order to counteract the higher 'tramp element' content arising from the use of poorer scrap, to add rare-earth metals to improve hot-workability. In the Crucible Steel Company's plant, with which the author is associated, all grades of austenitic and high-chromium steels which are going into bar stock are tapped at a relatively high temperature, held in the ladle for 10 minutes and poured at about 2870°F. (1575°C.).

Structure and Properties of 25-12 and 25-20 Chromium-Nickel Steels

D. J. CARNEY and E. R. ROSENOW: 'Effects of Chemical Composition and Heat-Treatment upon the Microstructure and Corrosion-Resistance of A.I.S.I. Types 309 and 310.'

Trans. Amer. Soc. Metals, 1955, vol. 48, *Preprint* 8; 21 pp.

The American Iron and Steel Institute specifications for 25-12 and 25-20 chromium-nickel steels call, respectively, for carbon not more than 0·20 and 0·25 per cent., and for material which is to be welded and subsequently used in service involving corrosive conditions a maximum of 0·08 per cent. carbon is usually laid down for both types of steel. This requirement is based on analogy with the use of low carbon in the 18–8 steels, for prevention of precipitation of intergranular chromium carbides during cooling after welding, and resulting impoverishment of corrosion-resistance.

The work described in this paper was carried out to determine the influence of carbon in steels of higher chromium and nickel content, with a view to ascertaining whether the present specification requirements are optimum.

The compositions of the steels examined are shown in the table below:—

It will be noted that for each grade a low (0·06%) and a high (0·16%) carbon level was included, and that in the 25-12 steel the nitrogen was varied from 0·04 to 0·19 per cent. In order to illustrate the effect of composition which would result in formation of considerable amounts of delta ferrite, a low-carbon steel of minimum nickel and high chromium was also included (the last composition in the table).

Annealing treatment usual for the types of steel studied comprises holding at 1950°F. (1065°C.) for half an hour, followed by water quenching, but it is known that in some steels falling within these specifications some chromium carbides may remain undissolved after such treatment, and the degree of carbide solution can have a marked influence on carbide precipitation during welding, cooling from annealing, or sensitization heat-treatment. In these experiments, therefore, two annealing treatments were used, one at a higher-than-normal temperature, to ensure complete solution of the carbides, the other at a low temperature chosen to secure dissolution of only 50 per cent. of the carbides present in the as-forged or as-rolled structure. After these solution treatments, specimens from each heat were (*a*) water quenched, (*b*) cooled at 1300°F. (722°C.) per hour, (*c*) cooled at 300°F. (167°C.) per hour. The cycles were chosen to approximate a range of cooling conditions which may exist in a welded structure.

Specimens which had undergone the various treatments were subjected to the boiling nitric acid and the boiling copper-sulphate/sulphuric acid tests, to determine susceptibility to intergranular corrosion. The structure of specimens after the respective heat-treatments was microscopically examined, with particular reference to carbide distribution.

In the 25-20 chromium-nickel steels (Type 310) excellent correlation was found between distribution of carbides and corrosion-resistance, as determined by both tests. Best resistance to corrosion after

A.I.S.I. Type	C %	Mn %	P %	S %	Si %	Cr %	Ni %	N %
310	0·06	1·57	0·008	0·005	0·78	24·56	21·54	0·085
310	0·16	1·52	0·007	0·008	0·73	25·68	21·29	0·058
309	0·06	1·56	0·009	0·024	0·84	22·44	14·05	0·069
309	0·06	1·58	0·006	0·006	0·50	21·23	15·03	0·080
309	0·05	1·56	0·008	0·006	0·65	23·26	14·04	0·190
309	0·16	1·50	0·010	0·020	0·75	24·39	14·29	0·042
309	0·16	1·50	0·010	0·006	0·54	23·41	14·06	0·069
309	0·15	1·58	0·007	0·006	0·64	23·68	14·16	0·170
309	0·06	1·47	0·010	0·007	0·75	25·00	12·04	0·089

slow cooling from the solution annealing temperature was associated with a microstructure containing a random distribution of undissolved carbides, which acted as nuclei for carbon precipitation. When the microstructure prior to solution annealing contained intergranular carbides, or when the carbides were completely dissolved during annealing, the steel became susceptible to intergranular corrosion after slow cooling from annealing. When the carbides were randomly dispersed and when the solution-annealing temperature was so low as to effect dissolution of only 50 per cent. of the carbides, the corrosion-resistance of the high-carbon (0·16%) 25-20 steel was equal to that of the low-carbon (0·06%) steel of the same type when both were slowly cooled from the annealing temperature.

In the 25-12 steels (Type 309) only fair correlation was found between carbide distribution and corrosion-resistance. Banding and segregation were more prevalent than in the more highly alloyed steel, and this affected correlation between grain-boundary carbides and resistance to corrosion. With nitrogen in a low or commercial range improved resistance was obtained by using a low solution-annealing temperature, resulting in only partial solution of randomly distributed carbides: this parallels observations made with the 25-20 steel. With a higher level of nitrogen, however, use of low solution temperatures did not greatly improve corrosion-resistance in steels slowly cooled after that treatment. At the low-carbon level increase in nitrogen was beneficial in enhancing corrosion-resistance, but this effect was not found in the 25-12 steels of higher carbon content. In the 25-12 steels, if rate of cooling from the solution treatment temperature is slow, resistance to intergranular corrosion can be much improved by adjusting the composition to ensure the presence of 20 per cent. of delta ferrite, thus minimizing intergranular carbide precipitation. In some cases, however, severe embrittlement was observed in steels containing these percentages of delta ferrite.

Corrosion by Hydrogen Sulphide at Elevated Temperatures

E. B. BACKENSTO, R. D. DREW and C. C. STAPLEFORD: 'High-Temperature Hydrogen-Sulphide Corrosion.' *Corrosion*, 1956, vol. 12, Jan., pp. 6t-14t; disc., pp. 14t-16t.

During recent years hydrogenation and dehydrogenation processes for upgrading petroleum fractions have become a commercial proposition in the petroleum industry. These processes operate at high temperatures and high hydrogen pressures, and there is usually formation of hydrogen sulphide, which is severely corrosive under working conditions. Although these facts were to some extent appreciated, it was known that in the oil industry low-chromium steels had been successfully used in plant employed in fractionation, catalytic and thermal cracking, and thermal reforming. When catalytic reformers for upgrading petroleum fractions were being designed it was therefore decided to use the low-chromium steels extensively: for example, such materials were used in the first Thermofor Catalytic Reformer which was put on stream in 1954 at the refinery of General Petroleum Corporation, at Torrance, California. High-sulphur naphthas were being treated, and on inspection of the plant after 42 days' operation it was found that deposits up to 0·1 in. thick had formed on all the steel parts, with the exception of some made from 18-8 chromium-nickel stainless steel.

In consequence of this experience, which clearly demonstrated the need for more reliable corrosion data applicable to this type of operation, a programme of research was initiated, to cover the study of resistance of various types of steel in hydrogen/hydrogen-sulphide atmospheres. The tests made, results of which are reported in this paper, covered exposure in commercial reforming units, and in laboratory pilot units using hydrogen, also laboratory experiments.

The materials used were representative of various grades of carbon steel, low- and medium-alloy chromium-molybdenum steels, high-chromium stainless steels, and chromium-nickel steels of the 18-8, 15-12, and 25-20 types. Specimens carrying aluminium coatings were also included.

The results of the various series of tests established that steels containing low percentages of chromium (up to about 5 per cent.) show little advantage over carbon steel. Of all the steels used, only the chromium-nickel types gave good resistance to attack over a wide range of temperature, pressure and hydrogen-sulphide concentration. Aluminium-coated steels also proved satisfactory.

The authors propose three methods for minimizing corrosion in service: (1) reduction of the hydrogen sulphide in the process streams; (2) use of chromium-nickel stainless-steel equipment; (3) protection by aluminium coatings.

Descaling of Steels and Alloys after Exposure to High-Temperature Water

R. FOWLER, D. L. DOUGLAS and F. C. ZYZES: 'Corrosion of Reactor Structural Materials in High-Temperature Water. I. Descaling Methods.'
General Electric Co., Knolls Atomic Power Laboratory Report KAPL-1198, 1954; 26 pp.+figures.

Metal oxide is usually formed on the surface of metallic materials exposed to high-temperature water, and in order to interpret the results of corrosion tests it is necessary to determine the amount of metal which has reacted. If the exact ratio of oxygen to metal in the metal-oxide film is known, and if all the scale remains on the exposed specimen, a simple 'weight-gain' determination will give the required information, but in most cases these ideal conditions do not exist. Films on commercial alloys are usually complex, and their composition will vary according to the aqueous medium in which the material has been exposed. In addition, there is, during dynamic tests, the possibility that some scale may be removed by erosion. Other means of determining the amount of metal reacted are therefore essential, and if quantitative removal of the scale ('descaling') can be effected, the difference between the weights of the specimen before and after test will supply the information.

This report comprises an account of experiments carried out to develop a method of descaling which, ideally, should be (1) capable of removing all types of scale from any type of metal, without attacking the base; (2) rapid; (3) independent of the length of exposure to the descaling medium and of its temperature; (4) relatively simple and inexpensive.

From the large number of descaling procedures described in the literature, methods based on the following principles were selected for examination:—mechanical removal by scrubbing, gas evolution at the metal surface, chemical action by inhibited acid, chemical action by inhibited molten caustic, chemical action by a strong reducing agent. Extensive details are given of the results of the various tests, made on carbon and low-alloy steels, a range of austenitic nickel-chromium stainless steels, high-chromium steel, nickel, and nickel-base alloys.

The three methods found most satisfactory for removal of scale formed in high-temperature water were:—

(1) Cathodic treatment at 45°C. in 5% sulphuric-acid solution containing, as inhibitor, 0·2% quinoline ethiodide: current density 15 amp./dm².

(2) Immersion in Clarke's solution (containing hydrochloric acid 100 g., antimonious oxide 2 g., stannous chloride 5 g.).

(3) Treatment by the sodium-hydride method.

Using the range of materials mentioned above, it was found that each of these methods has advantages and each has limitations. The cathodic treatment in sulphuric-acid solution descales many materials satisfactorily, especially carbon and low-alloy steels, but it is not effective for nickel and high-nickel alloys. Immersion in Clarke's solution descales carbon and low-alloy steels, and is an easy method, but this procedure also is unsuitable for high-alloy materials. The sodium-hydride procedure is applicable to the widest range of materials and, in general, this method can be used for removal of scales which cannot be removed by other treatments.

Cleaning of Stainless-Steel Food-Service Equipment

R. E. PARET: 'How to Clean Stainless Steel.'
Reprint from *Restaurant Equipment Dealer*, 1955, Sept.; 4 pp.

Issued by AMER. IRON AND STEEL INST., NEW YORK, 1955.

This is an essentially practical article, making recommendations for maintenance of stainless-steel equipment used for culinary and food-service equipment. Two basic rules are laid down: (1) clean frequently, and on a fixed schedule, and (2) select the simplest method.

For removal of ordinary dirt and food residues from surfaces operating at normal temperatures, soap-and-water washing is adequate: cleaning action may be hastened, where necessary, by addition of soda ash, baking soda, borax, or commercial non-abrasive cleaning agents.

The same treatment is frequently sufficient to remove spatterings or condensed vapours which have 'baked' onto cooking vessels, but in some cases a gentle-to-vigorous polishing action may be required. In such cases a paste made with water and ammonia as the liquid, and magnesium oxide, finely powdered pumice, or French chalk is suitable, or proprietary cleaners specially made for use on stainless steel may be employed. A soft cloth should be used, and rubbing should be in the direction of the polishing marks on the steel. For more resistant soil, a stainless-steel scouring sponge or wool of the finest possible texture may be used. It is essential *not* to use common steel wool, scouring pads, scrapers, wire brushes, files or other steel tools, since these mar the stainless-steel surface and leave embedded on it small particles of iron, which will ultimately cause rusting.

Although accumulation of soil such as grease, food, oil, etc., may be kept down by constant hot-water flushing, it is desirable periodically to use a strong cleaning solution. The following are recommended as excellent for removal of grease, oil, and fatty acids, where swabbing or rubbing is not practicable:—

(*a*) 4–6 per cent. solution of sodium metasilicate, trisodium phosphate, sodium metaphosphate, sodium pyrophosphate, *or*

(*b*) 5–15 per cent. caustic soda, used hot or cold.

For non-water-soluble materials the following organic solvents are suggested: carbon tetrachloride, naphtha, trichlorethylene, acetone, kerosene, petrol, ether, alcohol or benzene. In using such cleaners due precaution must be taken against risk of fire.

Stubbornly adherent deposits of organic, mineral or carbonaceous substances may be removed by means of 10 or 20 per cent. nitric acid, but it is imperative that such a cleaner should not be brought into contact with any part of the equipment which is not of stainless steel.

After using any strong cleaner liberal flushing with water is essential

Darkened areas which sometimes appear on stainless-steel surfaces after heating in service may be removed with stainless-steel wool or scouring pad. The occurrence of heat tinting may be prevented by avoiding overheating or the application of heat to empty vessels, and by ensuring that heat is not concentrated on too small an area.

In sterilizing stainless-steel equipment, particular care must be given to thorough rinsing away of solutions containing chlorine compounds, such as potassium hypochlorite. Stainless steel is resistant to attack by such compounds for about two hours, but if they are left in contact for a longer period the compounds may break down and release free chlorine, or may hydrolize, to form hydrochloric acid. In either case, severe pitting of the steel may result. By keeping the sterilization period short, and thoroughly rinsing after such treatment, chlorine-containing media may safely be used.

Reactor Materials and Design

See abstract on p. 28.

Monel Buckets in Salt Driers

'New Type Conveyor Belt.'

Chemical Age, 1956, vol. 74, Feb. 4, p. 336.

A new type of conveyor belt recently introduced commercially has a cover of Hypalon rubber, one of the Du Pont synthetic materials. The belt is part of machinery which lifts hot salt from a direct-fired rotary drier to storage bins 27 ft. above the drier. This belt, thus operating in mechanically and thermally severe conditions, works seven days a week, carrying the salt through a sheet-metal enclosure which, while keeping the salt dry, maintains temperatures of 150°–260°C. Belts of material previously used had lives of not more than two to three months, due to embrittlement of the rubber, but the Hypalon covers have given satisfactory life for at least twice that period. It is noted that the 56 buckets in which the salt is conveyed are made from Monel.

Influence of Oxygen on Corrosion of Stainless Steel in Sodium

E. F. BATUTIS, S. J. RODGERS and J. W. MAUSTELLAR: 'The Influence of Oxygen on the Corrosion of Armco Iron and Type 347 Stainless Steel in 1000°F. (540°C.) Sodium.'

Mine Safety Appliances Company Report, 1954; 14 pp.

Metallurgical Abstracts, 1955, vol. 23, Dec., p. 317.

'Corrosion rate of the iron varied from 0·20 mg./cm²/mth. at 0·005 wt. per cent. oxygen to 60·0 mg./cm²/mth. at 0·066 wt. per cent. oxygen. Below 0·036 wt. per cent. oxygen the stainless steel showed a weight increase, and above this figure a weight loss. The corrosion rate varied from −1·6mg./cm²/mth. at 0·008 wt. per cent. oxygen to +2·4 mg./cm²/mth. at 0·080 wt. per cent. oxygen.'

Corrosion of Car Bodies: Resistance of Plain and Alloy Steels

J. A. EDWARDS: 'Corrosion of Car Bodies. A Résumé of American Investigations.'

Automobile Engineer, 1955, vol. 45, Dec., pp. 550–2.

This article contains a résumé of information contained in a paper by J. C. HOLZWARTH, R. F. THOMSON and A. L. BOEGEHOLD, recently presented to the *Society of Automotive Engineers*. The nature of the corrosion found on cars in service has been correlated with laboratory, cyclic-humidity and outdoor-exposure tests, in sheltered and unsheltered positions, and observations have been made with regard to the relative corrosion-resistance of plain and low-alloy steels. The nature of the corrosion products formed, their relative degree of protective power, and other factors are considered in relation to the feasibility of replacing rimmed steel by other types. On the basis of the evidence presented, the conclusion is drawn that replacement of rimmed steel by even the best of the low-alloy steels tested would not be justified on economic grounds. In cyclic-humidity conditions carbon has proved to be the most potent element in reducing corrosion, but since the poor forming characteristics of high-carbon steels would preclude their use for car bodies, a search for solution of the problem of corrosion-resistance is being sought in an additive or a combination of additives which, like carbon, will produce increased resistance to corrosion, without adversely affecting forming characteristics.

Stainless-Steel Equipment in Production of Pharmaceuticals

'Terramycin Manufacture in Britain.'

Chemical and Process Engineering, 1955, vol. 36, Dec., pp. 437–8.

This article gives a description of the deep-fermentation antibiotics plant which is operated at Richborough, Kent, by Pfizer, Ltd., the British subsidiary of Charles Pfizer and Company, Inc., U.S.A. Terramycin is the principal product of this £2½-million factory, which is now producing enough of the drug to supply all requirements in the U.K. and the sterling area. It is estimated that an immediate saving will thus be made of 5 million dollars per annum in imports from the U.S.A. The parent Company, which was the first to manufacture an antibiotic (penicillin) by deep fermentation, is still the largest manufacturer of penicillin in the world.

The article describes the various stages involved in making terramycin, and it is of interest to note how extensive is the use of stainless-steel equipment, to ensure the hygienic, sterile conditions which are of such primary importance.

A further example of the value of stainless steel in equipment manufacturing pharmaceutical products is provided by reference to its employment in tanks used for mixing of polio vaccine at the Glaxo Laboratories: see *News Chronicle*, 1956, Jan. 20, p. 1.

Nickel-containing Materials in Marine Engineering

See abstract on p. 40.

Resistance of Nickel/Rhodium-Plated Parts to Moist Ozone

See abstract on p. 33.

High-Manganese Stainless Steels

F. P. HUDDLE: 'Nickel Conservation with High-Manganese Stainless Steels.'

Metal Progress, 1955, vol. 68, Dec., pp. 100–3.

The article comprises a review of the requirements, in the U.S.A., for nickel in stainless steels, principally of the 18–8 type, with consideration of the extent to which the high-manganese, lower-nickel steels could usefully be employed. A large part of the information presented is culled from a report prepared by E. V. BENNET, at the request of the U.S. Department of Defense, for the Materials Advisory Board of the National Academy of Sciences and the National Research Council. Tables illustrating the report include one showing the composition limits for high-manganese and for conventional 18–8-type chromium-nickel steels currently used in the U.S.A., and a breakdown of consumption of 18–8 steels in fifteen industrial fields and in military uses.

Nomograph for Calculation of Corrosion

H. K. BASS and R. V. ANDREWS: 'Nomograph for Corrosion Rate Calculations.'

Corrosion, 1956, vol. 12, Jan., pp. 4t–5t.

Confusion liable to arise due to variation in expressions used for rate of corrosion has been to some extent clarified by nomographs and conversion tables (to some of which the present authors give reference), but recently adoption of the rate expression mils per year has been advocated, and it is with this form that the nomograph given in this article is concerned.

M.p.y. is related to the density (g./c.c.) of the specimen exposed, designated as D; the area exposed (in.), A; time of exposure (days), T; and weight loss of the specimen (g.), W. The equation is as follows:—

$$\text{M.p.y.} = \frac{22{,}289\,W}{DAT}$$

The authors demonstrate that this equation may be solved graphically by the nomograph given, and that the results are satisfactorily accurate. A typical problem involving calculation of corrosion rate of iron is worked out to demonstrate the validity of the method.

Determination of Metals in Oils and Petroleum Products

See abstract on p. 29.

Analysis of Thin Films

See abstract on p. 29.

Joining of Nickel and Nickel Alloys

See abstract on p. 39.

Heat-Treatment of Welded Alloy Steels

See abstract on p. 41.

Nickel-Alloy Hard Facing on Compressor Rods

'Spray-Welded Alloy Overlay Triples Part Life.'

Iron Age, 1956, vol. 177, Jan. 12, p. 83.

This brief note reports experience in the use of hard-faced reciprocating rods in hydrogen compressors working at 800 p.s.i. pressure. Rods of hard-chromium-plated carbon steel had previously given lives of only about six months, being unable to withstand the abrasive conditions resulting from contact with the rod packing, which picks up dirt and other particles. It was therefore decided to install, in the Corpus Christi refinery of the Delhi-Taylor Oil Corporation, rods faced at the critical positions with a Colmonoy nickel-chromium-boron alloy. This type of coating, which is applied by spray welding, has a hardness of R_c 56–61, and is characterized by a low coefficient of friction. It is readily machined. Since the hard-faced rods were put into commission, in May 1954, no replacement has been necessary: the rods are still operating, indicating a service life at least three times that of the chromium-plated rods previously used. A short note is given on the essential features of the spray-welding process.

Specification for Carbon and Alloy Steel Plate, Sheet and Strip

See abstract on p. 40.

Corrigendum

Nickel Bulletin, 1955, vol. 28. No. 12, p. 211.

Left-hand column 'Nickel Compounds in Detection of Formaldehyde', l. 6 read 'as little as 0·5 γ of formaldehyde can be detected.'

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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